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FIZIČKA I MEHANIČKA SVOJSTVA KONVENCIONALNOG GLAS-JONOMER CEMENTA SA UGRAĐENIM KATJONSKIM SUPSTANCAMA

PHYSICAL AND MECHANICAL PROPERTIES OF CONVENTIONAL GLASS IONOMER CEMENT INCORPORATED WITH CATIONIC SUBSTANCES

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Sažetak

Uvod: *Proučavan je efekat antimikrobnih sredstava benzalkonijum hlorid (BH) i cetilpiridinijum hlorid (CPH) na restauracijski glas-jonomer zubni cement Fuji IX.*
Cilj: *Cilj studije bio je da se proceni da li dodavanje antimikrobnih sredstava narušava fizička i mehanička svojstva komercijalnog GJC Fuji IX.*
Materijali i metode: *Tokom faze mešanja, dodavane su koncentracije 1%, 2% i 3% antimikrobnih sredstava BH i CPH, po težini cementa , i proučavani su različiti efekti . U većini uzoraka došlo je do blage promene vremena stvrdnjavanja . Za merenje čvrstoće na pritisak i oslobađanja jedinjenja korišćeni su uzorci prečnika 4 mm i visine 6 mm. Oslobađanje antimikrobnih jedinjenja je analizirano UV-vidljivom spektrofotometrijom na talasnoj dužini od 259 nm za CPH i 214 nm za BH, u dejonizovanoj vodi.*
Rezultati: *Dobijeni rezultati su pokazali da se oslobađanje odvija mehanizamom difuzije tokom prva 2-3 sata, a koeficijenti difuzije variraju u zavisnosti od koncentracije. Vrednosti se kreću od 1,97 k 10-14 -1,78 k 10-12 m2 s-1. Otpuštanje antimikrobnih jedinjenja prestalo je nakon sedam dana, a ukupno otpuštanje iznosilo je između 2,15 i 4,84% početnog dodatka aditiva.*
Zaključak: *Oba jedinjenja imaju mali uticaj na vreme vezivanja GJC. Smanjenje čvrstoće na pritisam nije statistički znača jno. Cementi koji su sadržali CHC (1 i 2%), bili su statistički značajno slabiji od onih koji sadrže BH (p <0,05). Oba antimikrobna jedinjenja pokazuju konstantno oslobađanje iz GJC sa vrednostima koje su direktno proporcionalne vremenu i koncentraciji.*

Ključne reči: *benzalkonijum hlorid; cetilpiridinijum hlorid; kontrolisano oslobađanje; difuzija; glas-jonomer*

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Abstract

Background: *The effect of the antimicrobial agents benzalkonium chloride (BC) and cetylpyridinium chloride (CPC) on the restorative glass ionomer tooth cement (GIC) Fuji IX was investigated.*
Aim of the study: *The aim of the study was to determine whether the addition of antimicrobial compounds impairs the physical and mechanical properties of the commercial GIC Fuji IX.*
Materials and Methods: *The concentrations of 1%, 2% and 3% of antimicrobial agentsBC and CPC, by weight of the cement, were added during the mixing phase and different effects were studied.In most samples, there was a slight change in setting time. Samples with 4 mm diameter and 6 mm height were used to measure compressive strength and release. The release of antimicrobial compounds was analysed by UV-visible spectrophotometry at a wavelength of 259 nm for CPC and 214 nm for BC, in deionized water.*
Results: *The obtained results showed that the release takes place through the diffusion mechanism in the first 2-3 hours, and the diffusion coefficients vary depending on the concentration. The values range is from* 1.97×10^{-14} - 1.78×10^{-12} $m^2 s^{-1}$*.Release of antimicrobial compound had ceased after seven days, with total release representing between 2.15 and 4.84% of the initial additive loading.*
Conclusion: *Both compounds have minor effect on the setting time of the GIC. The reduction of compressive strength is not statistically significant. CPC containing cements (1 and 2%), were statistically significantly weaker, than those containing BC (p<0.05).Both antimicrobial compounds have shown constant release from the GIC with values which are directly proportional both to the time and to the concentration.*

Key words: *benzalkonium chloride, cetylpyridium chloride, controlled release, diffusion, glass-ionomer*

Uvod

Glas-jonomer cementi (GJC) se koriste u restorativnoj stomatologiji više od četrdeset godina[1]. Koriste se na razne načine i u različite svrhe. Najčešće se koriste u dečjoj stomatologiji kao direktni restaurativni materijali, ali se koriste i kao lajneri i podloge[1,2]. U ortodonciji se mogu koristiti za cementiranje ortodontskih bravica[3], a proučavani su i kao eksperimentalni koštani cementi[4,5].

Glas-jonomer cementi se sastoje od osnovnog stakla u prahu i vodenih rastvora poli-kiselina, tipično poli (akrilne kiseline) koji se vezuju kiselinsko-baznom reakcijom[6]. Prah je kalcijum -fluoroaluminosilikatno staklo rastvorljivo u kiselini slično onom kod silikata ali sa većim odnosom aluminijum -silikat koji povećava njegovu reaktivnost sa tečnošću . Međutim, zabeležena su i druga eksperimentalna stakla[7].

Glas-jonomer cementi se smatraju kao najprihvatljiviji restaurativni materijali koji poseduju pozitivne karakteristike fluora značajnim u procesima remineralizacije i antimikrobnog delovanja[1,8]. Takođe, imaju sposobnost pufera organskih kiselina, poput mlečne kiseline[9]. Ove kiseline generisane oralnim mikroorganizmima utiču na mineralnu fazu zuba - proces poznat kao demineralizacija ili erozija tvrdih zubnih tkiva[10]. Glas-jonomer cementi, koji poseduju pozitivne karakteristike fluora za procese remineralizacije i antimikrobnog delovanja, ističu se kao najprihvatljiviji restaurativni materijali. Jedno od najvažnijih svojstava glas-jonomer cemenata je sposobnost sporog i stalnog oslobađanja jona fluora tokom dugog perioda[11]. Ovaj period se može povećati unošenjem fluora u prisustvu rastvorenog fluorida poreklom iz pasta za zube i/ili fluorisanih vodica za ispiranje usta[12].

Pored oslobađanja fluoridnih jona, GJC se potencijalno mogu koristiti kao osnova za kontrolisano oslobađanje drugih aktivnih antimikrobnih komponenti[13]. Najčešće analizirano antimikrobno sredstvo je hlorheksidin, opisan kao zlatni standard za antibakterijsku primenu[14]. Iako neki antimikrobni agensi imaju potvrđeni efekat na smanjenje kariogene flore pljuvačke kada se koriste u sistemima za ispiranje ili zubnim pastama, rezultati u vezi sa njihovom ugradnjom u glas-jonomer cemente su i dalje oskudni, osim nekoliko pokušaja ugradnje cetilpiridinijum hlorida[15], benzalkonijum hlorida[15] i natrijuma fusidate[16]. Razmatrano je nekoliko aspekata dodavanja ovih supstanci.

Introduction

Glass-ionomer cements have been used in restorative dentistry for more than forty years[1]. They are used in a variety of ways and for different purposes. They are most often used in paediatric dentistry as direct restorative materials, but they are also used as liners and bases[1,2]. In orthodontics they can be used to cement orthodontic braces3, and have also been studied as experimental bone cements[4,5].

Glass-ionomer cements are cements that consist of a basic powdered glass and an aqueous solutions of polyacid, typically poly(acrylic acid) which are set by an acid-base reaction[6]. The powder is an acid-soluble calcium fluoroaluminosilicate glass similar to that of silicate, but with a higher alumina-silicate ratio that increases its reactivity with liquid. However, other experimental glasses have also been reported[7].

The glass-ionomer cements distinguish themselves as most acceptable restorative materials possessing the positive characteristics of fluorine in the processes of remineralisation and antimicrobial action[1,8]. Also, they have the ability to buffer organic acids, such as lactic acid[9].These acids generated by the oral micro-organisms effect the mineral phase of the tooth – a process known as demineralization or eroding of hard dental tissues[10]. The glass-ionomer cements, possessing the positive characteristics of fluorine in the processes of remineralisation and antimicrobial action, distinguish themselves as the most acceptable restorative materials.

One of the most important properties of glass-ionomer cementsis the capacity for slow and sustainable release of fluoride ions during a long period[11]. This period can be increased by fluoride uptake in the presence of dissolved fluoride originating from toothpastes and/or fluoridated mouthwashes[12].

In addition to the release of fluoride ions, GICs can potentially be used as templates for the controlled release of other active antimicrobial components[13]. The most frequently analysed antimicrobial agent has been chlorhexidine, described as a golden standard for antibacterial application[14]. Although some antimicrobial agents have a confirmed effect in the reduction of the cariogenic salivary flora when used in rinses or toothpastes, the results regarding their incorporation in glass-ionomer cements are still scanty, except several attempts for incorporation of cetylpyridinium chloride[15], benzalkonium chloride[15] and sodium fusidate[16].

I cetilpiridinijum hlorid (CPH) i benzalkonijum hlorid (BH), koji su ugrađeni u GJC, pokazali su stabilno oslobađanjekao i značajna antimikrobna svojstva, ali su oslabili vreme vezivanja[15,17]. Reakcija vezivanja hlorheksidina takođe je zabeležena sa sličnim nalazima[14]. Natrijum-fusidat je još jedna supstanca za koju se pokazalo da se može osloboditi iz glas-jonomer cemenata, postupkom difuzije. U literaturi postoji vrlo mali broj podataka koji se odnose na efekat natrijum-fusidata na hemiju vezivanja ili na eventualnu čvrstoću na pritisak

Cilj studije: Cilj ove studije bio je da se dobiju jasniji nalazi dodavanja katjonskih antimikrobnih jedinjenja cetilpiridinijum hlorida i benzalkonijum hlorida glas-jonomer cementu.

Materijali i metode

Studije su izvedene na komercijalnom restorativnom glas-jonomernom cementu Fuji IX (GC, Japan). Korišćena sz antimikrobna jedinjenja cetilpiridinijum hlorid (Sigma-Aldrich, Dorset, Velika Britanija) i benzalkonijum hlorid (Fluka, Nemačka). Antimikrobna jedinjenja su prvo ugrađena u poliakrilnu kiselinu glas -jonomer cementa, a zatim je prah postepeno dodavan i mešan zajedno do potpunog zasićenja. Antimikrobno sredstvo je dodato u strogim delovima od 1, 2 i 3 % mase cementa. Prethodne analize su utvrdile da su koncentracije 1, 2 i 3% antimikrobnih sredstava jednake 0,0032 g, 0,0064 g i 0,0128 g GJC Fuji IX. Pored toga, izvedeni su eksperimenti na cementu bez aditiva, kao kontrola . Vremena vezivanja određena su za cement koji sadrži različite nivoe aditiva, kao i za cement bez aditiva , koristeći Gillmore iglu(masa od 28 g), kako je navedeno u ISO991718 (Slika 1).

Čvrstoća na pritisak utvrđena je pomoću cilindričnih uzoraka dimenzija prečnika 4 mm i visine 6 mm. Kompleti od pet takvih primeraka pripremljeni su pomoću kalupa od nerđajućeg čelika i naloženi u sveže pomešane cementne paste (Slika 2). Ivice su ravne stezanjem metalnih ploča pomoću G-stezaljke. Uzorci su očvršćavani u pećnici na 37 oC tokom 1 sata, zatim su uklonjeni iz kalupa i čuvani još 23 sata u vodi na 37 oC pre ispitivanja. Ispitivanje je izvršeno na Instron Universal Testing Machine (Model 1193, Instron Corp., USA), brzinom poprečne glave od 1 mm / min (Slika 3). Određivanje količine antimikrobnih sredstava izvršeno je pomoću spektrofotometra UV-vis VARIAN - Cari 50 Tablet (Slika 4).

Both cetylpyridinium chloride (CPC) and benzalkonium chloride (BC), which are incorporated into the GICs, have shown steady release as well as significant antimicrobial properties, but haveweakenedthesetting time[15,17]. The setting reaction of chlorhexidine has also been reported with similar findings[14]. Sodium fusidate is another substance which has also been shown to be capable of being released from glass-ionomer cements, through a diffusion process. There is a very low number of data in the literature referring to the effect of sodium fusidate on either the setting chemistry or eventual compressive strength.

Aim of study: The aim of this study was to obtain clearer findings from the addition of cationic antimicrobial compounds cetylpyridinium chloride and benzalkonium chloride in glass ionomer cement.

Materials and methods

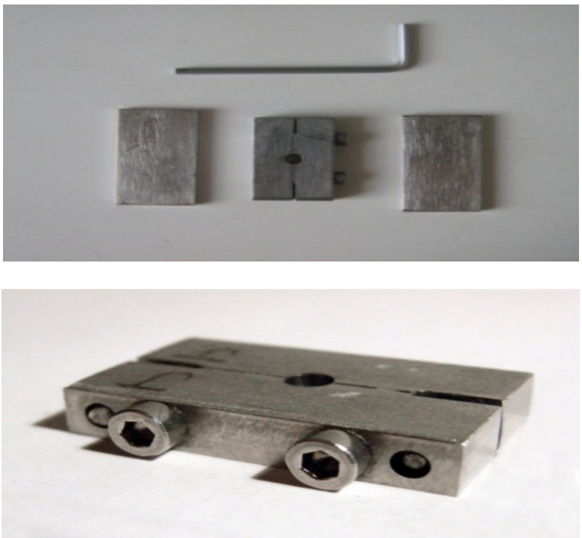
Studies were carried out using the commercial restorative grade glass-ionomer cement Fuji IX (GC, Japan). Antimicrobial compounds used were cetylpyridinium chloride (Sigma-Aldrich, Dorset, UK) and benzalkonium chloride (Fluka, Germany). The antimicrobial compounds were first incorporated into the glass ionomer cement's polyacrylic acid and then the powder was added gradually and mixed together until complete saturation. The antimicrobial agent was added in strict portions of 1, 2 and 3 % by mass of the cement. Preceding analyses had determined the concentrations of 1, 2 and 3 % of antimicrobial agents to be equivalent to 0.0032 g, 0.0064 g and 0.0128 g of GIC Fuji IX. In addition, experiments were carried out on additive-free cement, as controls. Setting times were determined for the cement containing the various levels of additive, as well as for cement without additives, using a Gillmore needle (28g mass), as specified in ISO991718 (Figure 1).

Compressive strength was determined using cylindrical specimens of dimensions 4 mm diameter x 6 mm height. Sets of five such specimens were prepared using stainless steel split moulds and loading them with freshly mixed cement pastes. Ends were made flat by clamping metal plates using a G-clamp. Specimens were cured in an oven at 37oC for 1 hour, then removed from the moulds, and stored for a further 23 hours in water at 37oC before testing. Testing was carried out on an Instron Universal Testing machine (Model 1193, Instron Corp., USA), with a cross-head speed of 1 mm/min (Figure 3). The determination of the amounts of the antimicrobial agents was done by the VARIAN

UV spektrofotometar je podešen na talasnu dužinu detekcije maksimalne apsorpcije (214 nm) za BH i 259 nm za CPH. Merenja su vršena na svakih 15 minuta tokom jednog sata, a zatim na 1, 2, 3, 4, 24 sata i 7 dana. Ucrtani su grafikoni Mt / M∞, a koeficijent difuzije je određen iz linearnog dela ovih grafikona, uzimajući nagib i zamenjuju ći u jednačinu D = s2 pl2 / 4. Podaci su analizirani na statističku značajnost pomoću Statisticaprograma.

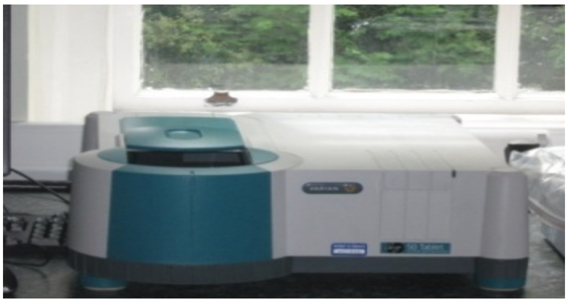
– Cary 50 Tablet UV-vis Spectrophotometer (Figure 4). The UV spectrophotometer was set to a detection wavelength of maximal absorption (214 nm) for BC, and 259 nm for CPC.The measurements were performed at every 15 min for an hour and then at 1, 2, 3, 4, 24 hours and 7 days. Graphs of Mt/M∞ were plotted, and the diffusion coefficient was determined from the linear portion of these graphs, taking the slope and substituting into the equation D = s2 πl2/4. Data were examined for statistical significance using the Statistica program.

Slika 1. Gilmorova igla
Figure 1. Gilmore needle



Slika 2. Metalni kalupi za pripremu uzoraka
Figure 2. Metal moduls for specimens preparation

Slika 3. Univerzalni stroj za testiranje "Instron" (model 1193, Instron corp., USA)
Figure 3. Universal Testing Machine (Instron Universal Testing Machine model 1193, Instron Corp., Canton, USA)



Slika 4. UV/vis. spektrofotometar VARIAN – Cary 50 Tablet
Figure 4. UV/vis. spectrophotometer VARIAN – Cary 50 Tablet

Rezultati

Učinak različitih koncentracija antimikrobnih sredstava na vreme stvrdnjavanja prikazan je u Tabeli 1. Nije bilo jasnih trendova, mada su postojali pokazatelji da je vreme vezivanja u nekim slučajevima blago produženo. To sugeriše da ovi aditivi imaju blagi inhibitorni efekat na reakciju vezivanja.

Podaci o čvrstoći na pritisak prikazani su u Tabeli 2. Ugrađivanje antimikrobnih jedinjenja u Fuji IX pokazalo je visoke vrednosti čvrstoće na pritisakkod cementa bez ugrađenog jedinjenja, koja je opadala sa porastom koncentracije ugrađenog aditiva. Ugrađivanje BH pokazalo je smanjenje vrednosti sa 146 MPa za 0% na 109,51 MPa za 3% jedinjenja. Kombinacija GJC-a i CPH-a dala je paradoksalne rezultate . Vrednosti dodataka od 1% i 2% su se kontinuirano smanjivale, dok su se drastično povećavale za 3% antimikrobnog jedinjenja.

Results

The effect of the different concentrations of antimicrobial agents on the setting time is shown in Table 1. There were no clear trends, though there was some indication that setting time was extended slightly in some cases. This suggests that these additives have a slight inhibitory effect on the setting reaction.

The data from compressive strength are shown in Table 2. The incorporation of the antimicrobial compounds into the Fuji IX, showed high values of compressive strength for the cement with no compound incorporated, which declined with the increase of the concentration. The incorporation of BCshoweddecreasing of values from 146 MPa for 0% down to 109.51 MPa for 3% of the compound. The combination of GIC with CPC, gave paradoxical results.

Tabela 1. Vreme stvrdnjavanja Fuji IX sa različitim nivoima dodavanja
Table 1. Setting time of Fuji IX with varying levels of addition

Aditiv/Additive	Vreme stvrdnjavanja/Setting time
None	4 min 35 s
1% BAC	4 min 40 s
2% BAC	4 min 30 s
3% BAC	4 min 30 s
1% CPC	4 min 38 s
2% CPC	4 min 45 s
3% CPC	4 min 25 s

Tabela 2. Prosečne vrednosti i statistička analiza pri određivanju tlačne čvrstoće GJC (vrednosti u MPa)
Table 2. Average values and statistical analysis when determining the compressive strength of GIC(values in MPa*)*

	Fuji IX + B.Chloride Prosek/average± (SD)	Fuji IX + CPC Prosek/average± (SD)
0%	146.29(8.57)	139.33(29.62)
1%	137.78(5.33)	90.10(10.79)
2%	119.72(10.78)	77.10(16.21)
3%	109.51(7.48)	126.04(4.14)
P	0.000010	0.000119
Tukey HSD test	Significant (p=0.000) 0% : 2% 0% : 3% 1% : 2% 1% : 3% 2% : 3%	Significant (p<0.05) 0% : 1% 0% : 2% 1% : 2% 2% : 3%

Ipak, prosečne vrednosti čvrstoće na pritisak bile su veće za kombinaciju Fuji IX - BC. Statistička analiza vrednosti (ANOVA) pokazala je postojanje statistički značajnih razlika u prosečnim vrednostima. Prema Tukei HSD testu, takođe su postojale statistički značajne razlike u prosečnim vrednostima za obe kombinacije.

Rezultati oslobađanja oba dodata antimikrobna sredstva prikazani su u Tabelama 3 i 4, kao i na Grafikonima 1 i 2. Analiza varijanse otpuštanja BH iz konvencionalnog Fuji IX GJC pokazala je statistički značajne razlike u prosečnim vrednostima za 1 i 2% antimikrobnog jedinjenja i beznačajne razlike za kombinaciju od 3%.

The values for the additions of 1% and 2% continually decreasedwhereas they drastically increased for 3% of the antimicrobial compound. Nevertheless, the average values of the compressive strength were higher for the combination Fuji IX - BC. The statistical analysis of the values (ANOVA) showed the existence of statistically significant differences in the average values. According to Tukey HSD test there were also statistically significant differences in the average values for both combinations.

The results from the release of both antimicrobial agents are shown in Tables 3 and 4 and in Graphs 1 and 2.

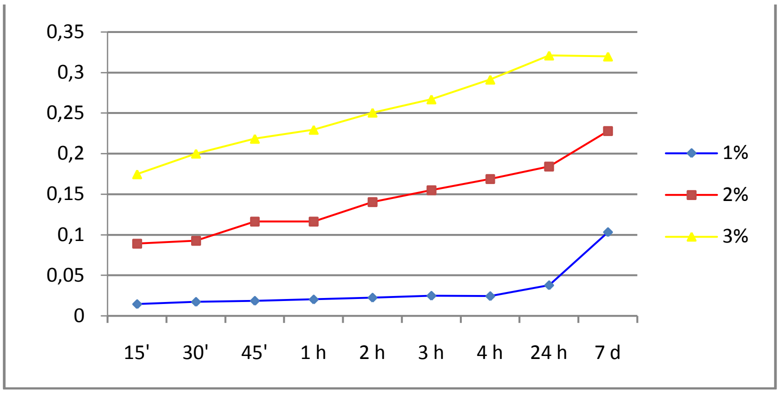
Tabela 3. Otpuštanje 1%, 2% i 3% cetilpiridinijum hlorida ugrađenog u Fuji IX (podaci dobijeni u jedinicama apsorbancije)
Table 3. Release of Cetylpyridinium Chloride 1%, 2% and 3%, incorporated in Fuji IX (data obtained in absorbance units)

Vreme/Time	Fuji IX + CPC 1% Prosek/average± (SD)	Fuji IX + CPC 2% Prosek/average± (SD)	Fuji IX + CPC 3% Prosek/average± (SD)
15 min.	0.01(0.00)	0.09(0.05)	0.17(0.10)
30 min.	0.02(0.00)	0.09(0.04)	0.20(0.11)
45 min.	0.02(0.00)	0.12(0.04)	0.22(0.11)
1 hour	0.02(0.00)	0.12(0.03)	0.23(0.11)
2 hours	0.02(0.00)	0.14(0.04)	0.25(0.10)
3 hours	0.02(0.00)	0.16(0.04)	0.27(0.10)
4 hours	0.02(0.00)	0.17(0.04)	0.29(0.10)
24 hours	0.04(0.01)	0.18(0.04)	0.32(0.10)
7 days	0.10(0.02)	0.23(0.05)	0.32(0.06)
P	0.000000	0.000009	0.000000
Tukey HSD test	Signifikantno/Significant (p<0.05) 7d.:15’,30’,45’,1,2,3.4,24h. 15’:24h. 30’:24h.	Signifikantno/Significant (p<0.05) 7d.:15’,30’,45’,1h.,2h. 15’:24h. 30’:24h.	Signifikantno/Significant (p<0.05) 7d.:15’,30’,45’,1,2,3h. 15’,30’:2,3,4,24h. 45’:24h.,7d.

Tabela 4. Otpuštanje 1%, 2% i 3% benzalkonijum hlorida ugrađenog u Fuji IX (podaci dobijeni u jedinicama apsorbancije)
Table 4. Release of Benzalkonium Chloride 1%, 2% and 3%, incorporated in Fuji IX(data obtained in absorbance units)

Vreme/Time	Fuji IX + Benzalkonioum Chloride 1% Prosek/average± (SD)	Fuji IX + Benzalkonioum Chloride 2% Prosek/average± (SD)	Fuji IX + Benzalkonioum Chloride 3% Prosek/average± (SD)
15 min.	0.25(0.06)	0.15(0.05)	0.17(0.10)
30 min.	0.26(0.06)	0.15(0.04)	0.20(0.11)
45 min.	0.23(0.05)	0.14(0.04)	0.29(0.11)
1 hour	0.28(0.06)	0.18(0.05)	0.23(0.11)
2 hours	0.29(0.06)	0.20(0.04)	0.25(0.10)
3 hours	0.28(0.06)	0.22(0.04)	0.27(0.10)
4 hours	0.29(0.06)	0.21(0.04)	0.29(0.10)
24 hours	0.35(0.05)	0.22(0.04)	0.32(0.10)
7 days	0.30(0.04)	0.28(0.04)	0.32(0.06)
P	0.040697	0.000012	0.162707
Tukey HSD test	Significant (p<0.05) 15’:24h. 45’:24h.	Significant (p<0.05) 7d.:15’,30’,45’,1h. 4h.:24h.	

Grafikon 1. Prosečne vrednosti 1%, 2% i 3% cetilpiridinijum hlorida ugrađene u Fuji IX tokom vremena
Chart 1. Average values na Cetylpyridinium Chloride 1%, 2% and 3%, incorporated in Fuji IX, over time



Grafikon 2. Prosečne vrednosti 1%, 2% i 3% benzalkonijum hlorida ugrađene u Fuji IX tokom vremena
Chart 2: Average values of released Benzalkonium Chloride 1%, 2% and 3%, incorporated in Fuji IX, over time

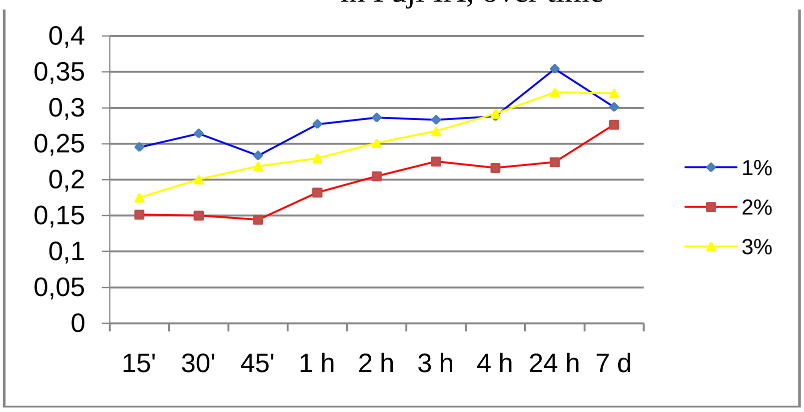


Tabela 5. Jednačine lenearne regresije i koeficijent korelacije za grafikone M t / M
Table 5. Linear regression equations and correlation coefficient for plots of Mt/M∞vs √t/s.

Aditiv (količina i tip)/ Additive (amount and type)	Jednaćina/Equation	Koeficijent korelacije/ Correlation coefficient
1% Benzalkonium chloride	$y = 2.607 \times 10^{-4}x + 0.038$	0.988
2% Benzalkonium chloride	$y = 1.585 \times 10^{-4}x + 0.047$	0.996
3% Benzalkonium chloride	$y = 4.208 \times 10^{-4}x + 0.066$	0.992
1% Cetylpyridinium chloride	$y = 1.53 \times 10^{-3}x + 0.181$	0.991
2% Cetylpyridinium chloride	$y = 1.078 \times 10^{-3}x + 0.218$	0.925
3% Cetylpyridinium chloride	$y = 8.858 \times 10^{-4}x + 0.173$	0.995

Tabela 6. Koeficijent difuzije aditiva na različitim nivoima dodavanja
Table 6. Diffusion coefficient of additives at various levels of addition

Aditiv (količina i tip)/ Additive (amount and type)	Koeficijent difuzije/ Diffusion coefficient ($m^2\ s^{-1}$)
1% Benzalkonium chloride	5.34×10^{-14}
2% Benzalkonium chloride	1.97×10^{-14}
3% Benzalkonium chloride	1.39×10^{-13}
1% Cetylpyridinium chloride	1.78×10^{-12}
2% Cetylpyridinium chloride	9.13×10^{-13}
3% Cetylpyridinium chloride	6.16×10^{-13}

Rezultati oslobađanja druge kombinacije (CPH i Fuji IX) pokazali su velike razlike u prosečnim vrednostima između analiziranih koncentracija. Oslobađanje se uglavnom odvijalo stabilno tokom istraženog perioda. Tokom tog vremenskog intervala, oslobađanje agenasa je bilo više ili manje uravnoteženo. Uzorci koji su sadržali veće koncentracije antimikrobnog jedinjenja, oslobodili su veću količinu jedinjenja. Ucrtavanje podataka u obliku Mt/M∞ dalo je prave linije tokom prva 2-3 sata, kao što je prikazano u Tabeli 5.

Jednačine linearne regresije i koeficijent korelacije za grafikone Mt / M∞ vs √t/s i koeficijent difuzije aditiva na različitim nivoima sabiranja prikazani su u Tabelama 5 i 6. Iako nije bilo određene sekvence u dobijenim rezultatima , najveća vrednost se dogodila za 3% GIC napunjenog BH. Suprotno tome, u kombinaciji Fuji IX i CPH, najveća vrednost se dogodila sa nivoom dodavanja od 1% i smanjila se redom od 1% do 3%.

The analysis of the variance from the release of BC from the conventional Fuji IX GIC showed statistically significant differences in the average values for 1 and 2% of the antimicrobial compound, and insignificant differences for the 3% combination. The results from the release of the other combination (CPCand Fuji IX)showed large differences in the average values between the analysed concentrations.

Release generally occurred steadily over the investigated period. During that time interval, the releasing of the agents was more or less equilibrated. The samples which contained higher concentrations of the antimicrobial compound, released larger amount of the compound. Plotting the data in the form of Mt/M∞ gave straight lines for the first 2-3 hours, as shown in Table 5.

Linear regression equations and correlation coefficient for plots of Mt/M∞ vs √t/s and diffusion coefficient of additives at various levels of addition are shown in Tables 5 and 6.

Although there was no specific sequence in the results obtained, the highest value occurred for a 3% GIC loaded with BC. By contrast, in the combination of Fuji IX and CPC, the highest value occurred with the 1% level of addition, and decreased in order from 1% to 3%.

Diskusija

Postoji niz studija koje opisuju efekat antimikrobnih jedinjenja na GJC[15-19]. U svim njima je zabeleženo poboljšanje antimikrobnih efekata GJC-a, ali takođe je primećeno i blago produženo vreme vezivanja i smanjenje čvrstoće na pritisak. Iako su proučavane razne vrste jedinjenja, čini se da je najviše proučavan hlorheksidin u različitim koncentracijama i kombinacijama (kombinacija sa diacetatom[14] i natrijum-fusidatom[16]). Takođe su proučavana kationska jedinjenja, posebno jedinjenja kvaternarnog amonijuma.

Sve druge vrste aditiva, manje ili više, inhibiraju vreme stvrdnjavanja GJC-a, kao što smo primetili. Smatra se da kvartarna amonijum jedinjenja, uključujući katjonska antimikrobna sredstva analizirana u ovom radu, inhibiraju vreme stvrdnjavanja interakcijom sa komponentom poli (akrilne kiseline)[20,21]. Međutim, inhibitorni efekat je primećen kod nekih drugih vrsta [22], kao što su metanol[22], 2-hidroksitil metakrilat[22] i natrijum hlorid[23]. Bez obzira na hemijsko poreklo, inhibicija vremena vezivanja je široko rasprostranjena i povezana je sa smanjenjem čvrstoće na pritisak . Ranije je zabeležen efekat kvaternarnih amonijumovih jedinjenja na čvrstoću na pritisak na konvencionalni glas-jonomer cement Fuji IX[17]. U toj studiji tom prahu je dodat CPH, a BH u tečnoj fazi cementa. Za razliku od uzoraka u ovoj studiji, bili su visoki 6 mm i 3 mm u prečniku. Iako je analiza rađena nakon 7 dana (uzorci su uskladišteni), za razliku od rada u trenutnoj studiji gde je čvrstoća na pritisak merena nakon 24 sata, čak i za nivo dodavanja od 1%, smanjenje čvrstoće je bilo izuzetno značajno ($p < 0,05$). Uočavanje značajnih razlika otežalo je i veliko standardno odstupanje u vrednosti čvrstoće na pritisak na uzorak cementa bez aditiva.

Oslobađanje antimikrobnih jedinjenja ugrađenih u GJC pojačava antibakterijsku aktivnost cementa[17]. Korišćenjem metode difuzije agara, ovo je eksperimentalno prikazano merenjem zone inhibicije oko cementnih diskova smeštenih u agar Petri posudama bakterijskih kultura[17]. U sadašnjem radu, oslobađanje BH i CPH mereno je direktno korišćenjem UV /vidljive spektrofotometrije. Analiza rezultata pokazala je da se rano oslobađanje antimikrobnih jedinjenja (2-3 sata) zasnivalo na difuziji, sa koeficijentima difuzije u opsegu. Ranije je pokazano da se oslobađanje nekih antimikrobnih jedinjenja, poput hlorheksidin-diacetata i natrijum-fusidata, javlja difuzijom u ranim fazama[14,16]. Koeficijenti difuzije natrijum-fusidata kretali su se između 3,0 i 4,4 k 10^{-12} m^2 s^{-1} i bili su

Discussion

There are a number of studies describing the effect of antimicrobial compounds on GIC[15-19] In all of them animprovement of the antimicrobial effects of GICs was noted, but slightly extended setting time and decrease in compressive strength were also noted. Although various types of compounds have been studied, chlorhexidine in different concentrations and combinations seems to be the most studied (the combination with diacetate[14] and sodium fusidate[16]).Cationic compounds, especiallyquaternaryammonium compounds, have also been studied.

All other types of additives, more or less inhibit the setting time of the GIC, as we have observed. Quaternary ammonium compounds, including the cationic antimicrobial agentsanalysed in this paper, are thought to inhibit thesetting time by interaction with the poly(acrylic acid) component[20,21]. However, an inhibitory effect has been observed in some other species, such as methanol[22], 2-hydroxytyl methacrylate[22]. and sodium chloride[23]. Regardless of chemical origin, inhibition of setting time is widespread, and is associated with a reduction in compressive strength.

The effect of quaternary ammonium compounds on compressive strength of the conventional glass-ionomer cement Fuji IX has been reported previously[17]. In that study, CPC was added to the powder, and BC was added in the liquid phase of the cement. Unlike the samplesin this study, they were 6 mm high x 3 mm in diameter. Although the analysis was done after 7 days (the samples were stored), in contrast to the work in the current study where the compressive strength was measured after 24 hours, even for the 1% level of addition, reduction in strength was highly significant ($p<0.05$). Observing significant differences was also hampered by the large standard deviation in the compressive strength value for the cement sample without additive.

The release of antimicrobial compounds incorporated into the GIC enhances the antibacterial activity of cements[17].

Using the agar diffusion method,this was shown experimentally by measuring the zone of inhibition around cement discs placed in agar Petri dishes of bacterial cultures[17]. In the current work, release of BC and CPCwas measured directly using UV/visible spectrophotometry. The analysis of the results showed that the early release of antimicrobial compounds (2-3 hours) was based on diffusion, with diffusion coefficients in the range.

nešto viši od onih koji su navedeni u BH i CPH u ovoj studiji. Ukupno oslobađanje antimikrobnih sredstava dobijenih u ovoj studiji bilo je između 2 i 5% od početne koncentracije, za razliku od natrijum fusidata koji je otpustio koncentraciju oko 20-22% od početnog opterećenja[16].

Rezultati dobijeni u ovoj studiji potvrđuju korisnost GJC-a kao potencijalnih materijala sa kontrolisanim oslobađanjem, posebno za antimikrobna sredstva. Modifikovani GJC mogu se široko koristiti u tehnici atraktivnog restorativnog tretmana u nerazvijenim zemljama[19] ili kod pacijenata sa invaliditetom i pacijenata sa posebnim potrebama čija je oralna higijena ugrožena.

Zaključak

Obe antimikrobne supstance imaju samo manje efekte na vreme vezivanja, tj. reakcija vezivanja je u određenoj meri inhibirana.

Utvrđeno je da je čvrstoća na pritisak u kontrolnoj grupi mnogo niža od tvrdnje proizvođača Čvrstoća na pritisak se smanjivala sa porastom koncentracije antimikrobnih jedinjenja, ali ne u statistički značajnoj meri.

Uzorci koji sadrže CPH od 1 i 2% bili su slabiji do statistički značajne mere od onih koji sadrže BH ($p < 0{,}05$).

Antimikrobna jedinjenja BH i CPH neprekidno su se oslobađala iz glas-jonomer cemenata sa vrednostima koje su bile direktno proporcionalne vremenu i koncentraciji . Ova jedinjenja su puštena u dejonizovanu vodu pomoću mehanizma difuzije tokom prva 2-3 sata. Koeficijenti difuzije varirali su u zavisnosti od koncentracije i kretali su se u opsegu 1,97 k 10^{-14} -1,78 k 10^{-12} m^2 s^{-1}. Ukupno oslobađanje variralo je sa koncentracijom i bilo je vrlo nisko, tj. između 2,15 i 4,84% početnog dodatka aditiva.

Previously, the release of some antimicrobial compounds such as chlorhexidine diacetate and sodium fusidate has been shown to occur by diffusion in the early stages[14,16]. The diffusion coefficients for sodium fusidate ranged between 3.0 and 4.4 x 10^{-12} m^2 s^{-1}, and wereslightly higher than those specified for BC and CPC in this study.Total release of antimicrobial agents obtained in this study was between 2 and 5% from the initial concentration,unlike sodium fusidate which released concentration was at around 20-22% of initial loading[16].

The results obtained in this study confirm the usefulness of GICsas potential controlled release materialsespecially for antimicrobial agents. Modified GICs can be widely used in Atraumatic Restorative Treatment technique in underdeveloped countries[19], or in patients with disabilities and special needspatients whose oral hygiene is compromised.

Conclusion

Both antimicrobial substances have only minor effects on the setting time, i.e., the setting reaction was inhibited to an extent.

The compressive strength in the control group was found to be much lower than the manufacturer's claim.The compressive strength decreased with the increase in the concentration of the antimicrobial compounds, but not to a statistically significant extent.

Specimens containing CPC at 1 and 2% were weaker to statistically significant extent than those containing BC ($p<0.05$).

The antimicrobial compounds BC and CPCwere continually released from the glass-ionomer cements with values which were directly proportional both to the time and to the concentration. These compounds were released into deionized water by a diffusion mechanism for the first 2-3 hours. Diffusion coefficients varied with concentration and were in the range 1.97 x 10^{-14} -1.78 x 10^{-12} m^2 s^{-1}. Total release varied with concentration, and was very low, i.e. between 2.15 and 4.84% of the initial additive loading.

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