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IZVORNI ZNANSTVENI RAD

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The Impact of Different Irrigation Protocols on the Bond Strength of Self-Adhesive Resin Cement to Radicular Dentin

Utjecaj različitih irigacijskih protokola na snagu veze samoadherirajućega kompozitnog cementa na radikularni dentin

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Abstract

Objective: The objective of the study was to investigate the influence of different irrigation protocols on the shear bond strength (SBS) of self-adhesive resin cement (SARC) on primed radicular dentin. **Methods:** Radicular dentin slabs (N=58) were embedded in acrylic. Subsequently, they were polished and randomly assigned to five experimental groups (N=8-12) and one control group, CG (N=8). Irrigation solutions used were 2.5% sodium hypochlorite (H), 17% ethylenediamine tetraacetic acid (EDTA), 9% etidronic acid with hypochlorite (HEDP), 0.9% saline solution (SS), and 2% chlorhexidine (CHX). Each experimental group underwent a different irrigation protocol for two minutes: 1) H/EDTA/H, 2) HEDP, 3) H/EDTA/CHX, 4) H/EDTA/SS and 5) HEDP/SS. The CG samples were not treated with any irrigation solution. After drying, the adhesive-enhancing primer (AEP) and dual-curing SARC were applied to the radicular dentin using molds corresponding to the Ultra Tester (Ultradent Products, South Jordan, USA). The specimens were stored at 37 °C in distilled water and broken after 10 days in shear mode. The results were analyzed using ANOVA and post-hoc Games-Howell test, α=0.05. **Results:** CG exhibited the highest SBS. With the exception of HEDP and HEDP/SS, all experimental groups showed significantly lower SBS compared to CG ($p<0.05$). **Conclusions:** HEDP appears to be an adequate solution for cleaning the root canal prior to dentin priming and fiber post cementation with SARC.

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Introduction

Timely and adequate post-endodontic care is crucial for the preservation of endodontically treated teeth (1). In cases of extensive loss of crown structure, glass fiber posts cemented with composite cements are a common therapeutic choice due to their satisfactory survival rates and biomimetic behavior (2, 3). Optimal post placement combined with reliable adhesion is a prerequisite for effective sealing and the prevention of micro- and nano-leakage (2, 4). The factors that influence the adhesion of glass fiber-reinforced composite posts to radicular dentin are related to the substrate (dentin), the post and the luting agent (cement) (2, 3, 5-7). Thus, the overall

Uvod

Pravodobna i adekvatna postendodontska opskrba ključna je za očuvanje endodontski liječenih zuba (1). U slučaju opsežnog gubitka krune zuba, vlaknima ojačane kompozitne nadogradnje cementirane kompozitnim cementom čest su terapijski izbor zbog zadovoljavajuće stope preživljavanja i biomimetičkog ponašanja (2, 3). Optimalno postavljanje nadogradnje, u kombinaciji s adekvatnom adhezijom, preduvjet je za učinkovito brtvljenje i sprječavanje mikro i nanopropuštanja (2, 4). Čimbenici koji utječu na adheziju kompozitnih nadogradnji ojačanih staklenim vlaknima na radikularni dentin povezani su s podlogom (dentin), nadogradnjom i bond strength of the glass fiber post to dentin is determined by the post–cement interface and the cement–dentin interface (8-11). The interaction of the cement with the radicular dentin on the one hand and the post on the other is influenced by the composition of the cement and its curing kinetics (3). In particular, achieving good adhesion to radicular dentin remains a major clinical challenge despite advances in polymerization and chemistry of resin cements (7, 8). In addition, several studies show that adhesion failures occur predominantly at the dentin-cement interface, which is the weakest link in terms of bond strength (9-11). Debonding is the main cause of failure of glass fiber-reinforced composite posts, which is due to the sensitivity of the cementation technique involving sealer and gutta-percha removal, dentin disinfection, smear layer removal, and moisture control (3, 7). The simplification of cementation techniques through the use of self-adhesive resin cements (SARC) offers advantages due to a reduced number of phases and technical sensitivity (12, 13). These types of cements contain multipurpose acids and methacrylate monomers with a phosphate group, and these contents should eliminate the need for prior use of adhesion systems (12, 13). However, G-CEM One (GC, Tokyo, Japan) is a luting material consisting of self-adhesive cement and G Cem One Adhesive Enhancing Primer (AEP, GC, Tokyo, Japan). The primer contains functional monomers and a chemical initiator that accelerates the chemical curing of the cement from the tooth surface, thus improving the bond between cement and dentin (13,14).

Root canal treatment involves chemomechanical preparation, which means that instrumentation is accompanied by irrigation. After completion of instrumentation, the root canal space is irrigated with a sequence of disinfectant and chelating solutions according to one of several final irrigation protocols (15-17). The most commonly used final irrigation protocols in the literature were NaOCl/EDTA/NaOCl, followed by NaOCl/EDTA/CHX with duration of 90-120 seconds, with or without agitation or ultrasonic activation (15, 16, 18). The cleanliness of the dentin surface is also important for adequate interaction with the cement and the success of treatment with glass fiber posts (8, 10). Cleaning and disinfection of the post space, with or without removal of the smear layer, may also influence bond strength. The most frequently used solutions for cleaning and disinfecting the post space are also sodium hypochlorite (NaOCl), chlorhexidine (CHX), ethylenediaminetetraacetic acid (EDTA) and a combination of these liquids (10,19-21). There is no unanimous opinion on the order in which NaOCl and EDTA should be used for cleaning and disinfection (22). According to some authors, it is not advisable to alternate NaOCl and EDTA since this could compromise the primary effect of both rinsing fluids (21, 23). Some authors suggest using NaOCl before EDTA, as NaOCl causes erosion on dentin when used after the removal of smear layer by EDTA (24). On the other hand, EDTA leaves the organic matrix of dentin exposed, and NaOCl rapidly degrades the exposed organic components of dentin (25, 26). The reduced collagen component due to the proteolytic activity of NaOCl may have a positive effect on the resistance of the future hybrid layer to degrada-

vezujućim sredstvom (cementom) (2, 3, 5 – 7). Zato je ukupna snaga adhezijske veze kompozitne nadogradnje pojačane staklenim vlaknima na dentin određena spojem između nadogradnje i cementa te spojem cementa i dentina (8 – 11). Na interakciju cementa s radikularnim dentinom s jedne strane, i nadogradnjom s druge strane, utječe sastav cementa i njegova kinetika stvrdnjavanja (3). U prvom redu klinički izazov ostaje postići dobru adheziju na radikularni dentin, unatoč napretku u polimerizaciji i kemiji kompozitnih cemenata (7, 8). Uz to, u nekoliko studija autori pokazuju da se neuspješna adhezija pojavljuje pretežno na granici dentina i cementa, što je najslabija karika kad je riječ o snazi adhezijske veze (9 – 11). Odcementiranje kompozitnih nadogradnji ojačanih vlaknima glavni je uzrok neuspjeha terapije, a uzroci takva ishoda su u osjetljivosti tehnike cementiranja koja uključuje uklanjanje paste i gutaperke, dezinfekciju dentina, uklanjanje zaostatnoga sloja i kontrolu vlage (3, 7). Prednost pojednostavljene tehnike cementiranja uporabom samoadherirajućih kompozitnih cemenata (SARC) manji je broj faza i manja tehnička osjetljivost postupka (12, 13). Ta skupina cemenata sadržava višenamjenske kiseline i metakrilatne monomere s fosfatnom skupinom koji omogućuju izostavljanje uporabe adhezijskih sustava (12, 13). Međutim, G-CEM One (GC, Tokio, Japan) materijal je koji se sastoji od samoadherirajućega kompozitnog cementa i primera za poboljšanje adhezije G Cem Onea (AEP, GC, Tokio, Japan). Primer sadržava funkcionalne monomere i kemijski inicijator koji ubrzava kemijsko stvrdnjavanje cementa pri površini zuba, čime se poboljšava veza između cementa i dentina (13, 14).

Liječenje korijenskog kanala uključuje kemomehaničku pripremu, što znači da je instrumentacija kanala popraćena njihovom irigacijom. Poslije završene instrumentacije, irigacija prostora korijenskog kanala obavlja se nizom dezinficirajućih i kelatorskih otopina prema jednome od nekoliko završnih protokola irigacije (15 – 17). Najčešće korišteni protokoli završnoga ispiranja u literaturi bili su NaOCl/EDTA/NaOCl, a zatim NaOCl/EDTA/CHX u trajanju od 90 do 120 sekunda, s agitacijom ili ultrazvučnom aktivacijom, ili bez agitacije ili ultrazvučne aktivacije (15, 16, 18). Čistoća površine dentina također je važna za odgovarajuću interakciju s cementom i uspjeh terapije kompozitnim kolčićima ojačanima staklenim vlaknima (8, 10). Čišćenje i dezinfekcija prostora za kolčić, s uklanjanjem zaostatnoga sloja ili bez toga, također može utjecati na snagu adhezijske veze. Najčešće korištena sredstva za čišćenje i dezinfekciju prostora jesu natrijev hipoklorit (NaOCl), klorheksidin (CHX), etilendiamintetraoctena kiselina (EDTA) i kombinacija tih tekućina (10,19 – 21). Ne postoji jednoglasno mišljenje o redoslijedu kojim bi se NaOCl i EDTA trebali upotrebljavati za čišćenje i dezinfekciju (22). Prema stajalištima nekih autora, ne preporučuje se naizmjenično primjenjivati NaOCl i EDTA-u, jer bi to moglo ugroziti primarni učinak pojedine tekućine za ispiranje (21, 23). Drugi predlažu korištenje NaOCl-a prije EDTA-e, jer NaOCl uzrokuje eroziju dentina kada se koristi poslije uklanjanja zaostatnog sloja EDTA-om (24). S druge strane, EDTA ostavlja organski matriks dentina eksponiranim, a NaOCl brzo razgrađuje izložene organske komponente dentina (25, 26). Smanjeni udio kolagena, tion (25, 26). Recently, 1-hydroxyethylidene-l, l-diphosphonic acid (HEDP), also known as etidronic acid or etidronate, has been increasingly investigated as an irrigation solution in endodontics (21-23, 27). HEDP acts as a mild chelating agent and is a promising substitute for EDTA (22, 23). One of its remarkable properties is its compatibility with sodium hypochlorite, allowing it to be mixed without compromising its antimicrobial efficacy. Most used at a concentration of 9 % with 2.5 % NaOCl, HEDP exhibits a demineralization effect less potent than EDTA (21,23,27). In the context of strategies for collagen reduction/depletion in dental adhesion, the less strong demineralization could have a positive effect on the bond strength of dental materials to dentin (28).

Previous studies investigating the influence of irrigants on fiber-post bond strength mainly focused on the application of a single solution in the post-space (10,29). The aim of the present study was to investigate the effects of different disinfectant and chelating solutions applied in different sequences (NaOCl, CHX, EDTA and etidronic acid) on the bond strength between G Cem One SARC and dentin primed with G Cem One Adhesive Enhancing Primer (AEP, GC, Tokyo, Japan). The effects of irrigation with etidronic acid on the bond strength of root canal sealers (30,31) and some SARCs has been studied (32,33) but no acidic primers with chemical initiator were used prior to SARC application in the aforementioned studies. The null hypothesis was that different irrigation protocols would have no effect on the bond strength of G Cem One SARC on primed radicular dentin.

zbog proteolitičke aktivnosti NaOCl-a, može povećati otpornost budućega hibridnoga sloja na razgradnju (25, 26). U posljednje se vrijeme sve češće istražuje 1-hidroksietiliden-l,l-difosfonska kiselina (HEDP), poznata i kao etidronska kiselina ili etidronat, kao sredstvo za ispiranje u endodonciji (21 –23, 27). HEDP djeluje kao blago kelacijsko sredstvo i obećavajuća je zamjena za EDTA-u (22, 23). Jedno od njegovih izvrsnih svojstava jest kompatibilnost s natrijevim hipokloritom, što mu omogućuje miješanje bez ugrožavanja njegove antimikrobne učinkovitosti. U najčešće korištenoj koncentraciji od 9 % s 2,5 % NaOCl-a, HEDP pokazuje manje izraženi demineralizacijski učinak od EDTA-e (21, 23, 27). U kontekstu nastojanja da se smanji udio kolagena u intersticijskom vezujućem sloju, smanjena demineralizacija mogla bi pozitivno utjecati na vezivanje dentalnih materijala na dentin (28).

U dosadašnjim studijama u kojima se istraživao utjecaj sredstava za ispiranje na snagu veze kompozitnih nadogradnji uglavnom se ispitivala primjena jedne otopine u intraradikularnom prostoru (10, 29). Svrha ovog istraživanja bila je istražiti učinke različitih dezinfekcijskih otopina i kelatora primijenjenih različitim redoslijedom (NaOCl, CHX, EDTA i HEDP) na snagu veze između G Cem One SARC-a i dentina prije toga tretiranoga G Cem One Adhesive Enhancing Primerom (AEP, GC, Tokio, Japan). Ispitivani su učinci irigacije etidronskom kiselinom na snagu veze pasti za punjenje (30, 31) i nekih SARC-a (32, 33), ali u navedenim studijama nisu korišteni kiseli primeri s kemijskim inicijatorom prije same primjene SARC-a. Nulta hipoteza bila je da različiti protokoli irigacije neće utjecati na čvrstoću adhezivne veze G Cem One SARC-a na radikularnom dentinu prije toga tretiranom primerom.

Materials and methods

The study was approved by the Ethics Committee of the School of Dental Medicine, University of Zagreb, approval number 05-PA-30-16-3/2023.

Preparation of dentin substrate

Forty non-carious human third molars with fully developed roots were collected and preserved in 1% chloramine solution for a period of one month after extraction. The crowns of the teeth were sectioned with a fissure diamond bur at the enamel-cement junction. From the coronal third of the roots, 2.2 mm thick dentin slabs were cut with a low-speed saw (IsoMet, Bühler, Düsseldorf, Germany). The saw ran perpendicular to the longitudinal axis of the roots at 200 revolutions per minute with continuous water cooling. The radicular dentin slabs were then stored in saline solution until they were mounted in cold-curing methacrylate resin (Technovit 4004, Kulzer, Germany). The samples with insufficient dentin width were excluded (<2.3 mm). An Ultradent mold (Ultradent Products, South Jordan, UT, USA) was used to embed radicular dentin specimens in the methacrylate resin. To create a flat bonding surface, the dentin surface was polished with 600 grit silicon carbide (SiC) paper (PRESI, Eybenes, France), rinsed thoroughly and stored in distilled water until further testing.

Materijali i postupci

Istraživanje je odobrilo Etičko povjerenstvo Stomatološkog fakulteta Sveučilišta u Zagrebu –broj odobrenja 05-PA-30-16-3/2023.

Priprema dentinskih uzoraka

Četrdeset intaktnih humanih trećih kutnjaka s potpuno razvijenim korijenima prikupljeno je i čuvano mjesec dana nakon ekstrakcije u 1-postotnoj otopini kloramina. Krunice zuba bile su prerezane fisurnim dijamantnim svrdlom na spoju cakline i cementa. Dentinske ploče debljine 2,2 mm izrezane su iz koronarne trećine korijena pilom male brzine (IsoMet, Bühler, Düsseldorf, Njemačka) usmjerenom okomito na uzdužnu os korijena pri 200 okretaja u minuti, uz kontinuirano hlađenje vodom. Ploče radikularnoga dentina pohranjene su zatim u fiziološkoj otopini dok nisu ugrađene u hladnopolimerizirajuću metakrilatnu smolu (Technovit 4004, Kulzer, Njemačka). Isključeni su uzorci s nedovoljnom širinom dentina (< 2,3 mm). Kalup Ultradent (Ultradent Products, South Jordan, UT, SAD) korišten je za ugradnju uzoraka radikularnoga dentina u metakrilatnu smolu. Da bi se dobila glatka ploha za adheziju, površina dentina ispolirana je brusnim papirom sa silicijevim karbidom (SiC) granulacije 600 (PRESI, Eybenes, Francuska), temeljito isprana i pohranjena u destiliranoj vodi do daljnjeg ispitivanja.

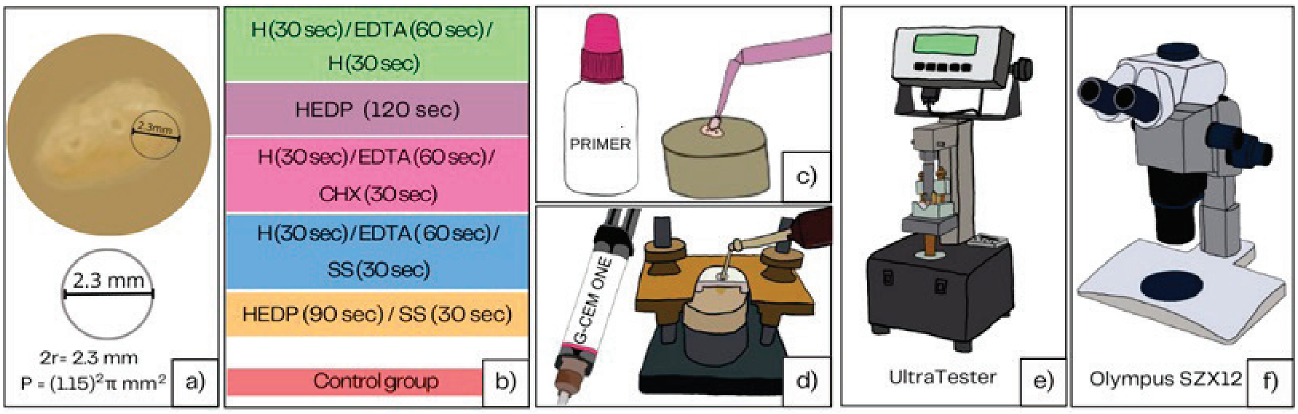

Figure 1 Preparation and analysis of samples : a) dentine slabs prepared form radices of intact third molars mounted in acrylic resin; b) different irrigation protocols in five experimental groups and control group; c) Application of the Primer on the area defined by a hole punctured in self adhesive strip; d) Application of the SARC using UltraTester mold; e) Shear bond strength testing; f) Fracture analysis. EDTA- ethylene-diamine-tetra-acetic acid, H-sodium hypochlorite, SS - saline solution, HEDP - etidronic acid, SARC – self adhesive resin cement.

Slika 1. Priprema i analiza uzoraka: a) dentinski uzorci pripremljeni od korjenova intaktnih trećih kutnjaka uloženih u akrilnu smolu; b) različiti protokoli irigacije u pet eksperimentalnih skupina i kontrolnoj skupini; c) nanošenje primera na područje definirano rupom probušenom u samoljepljivoj vrpci; d) primjena SARC-a s pomoću UltraTester kalupa; e) Ispitivanje čvrstoće smične veze; f) analiza frakture. EDTA – etilendiamintetraoctena kiselina, H – natrijev hipoklorit, SS – fiziološka otopina, HEDP – etidronska kiselina, SARC – samoadherirajući kompozitni cement

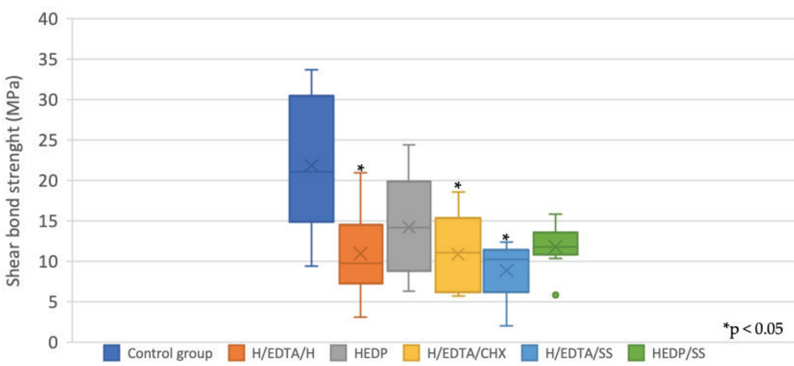

Figure 2 Differences in arithmetic means of SBS in groups treated with different irrigation protocols. Control group exhibited the highest mean SBS. Groups H/EDTA/H, H/EDTA/CHX and H/EDTA/SS exhibited significantly lower SBA that the CG (marked with *). In HEDP and HEDP/SS groups SBS was not statistically significantly lower than in the CG. EDTA- ethyle-ne-di mine-tetra-acetic acid, H-sodium hypochlorite, SS - saline solution, HEDP - etidronic acid.

Slika 2. Razlike u aritmetičkim sredinama SBS-a u skupinama tretiranima različitim protokolima irigacije. Kontrolna skupina pokazala je najvišu srednju vrijednost SBS-a. Skupine H/EDTA/H, H/EDTA/CHX i H/EDTA/SS pokazale su značajno niži SBA od CG-a (označenog s *). U HEDP i HEDP/SS skupinama SBS nije bio statistički značajno niži nego u CG-u. EDTA – etilennedimintetraoctena kiselina, H – natrijev hipoklorit, SS – fiziološka otopina, HEDP – etidronska kiselina

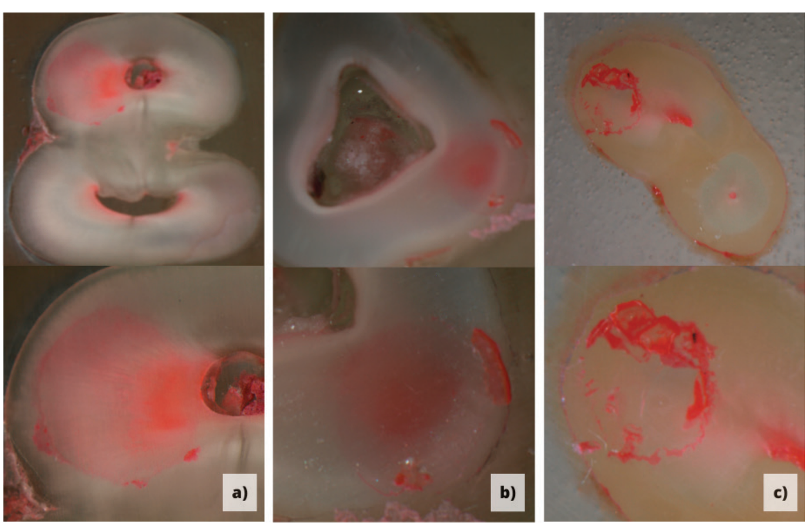

Figure 3 Representative images of; a) adhesive failure (AD), b) and c) mixed failures (MF) with different proportions of adhesive and cohesive failure in cement.

Slika 3. Reprezentativne slike analize fraktura: a) adhezivni lom (AD), b) i c) mješoviti lom (MF) s različitim omjerima adhezivnoga i kohezivnoga loma u cementu

Irrigation and bonding procedure

A total of 72 samples were prepared and randomly divided into 6 groups: five experimental and one control group (Figure 1). Some samples were lost during the bonding process because the slot in the mold did not fit perfectly into the hole in the polymer adhesive strip, hence the number of samples in the different groups varied between 8 and 12. Irrigation solutions used were 2.5% sodium hypochlorite solution sodium hypochlorite solution (H), ethylenediamine tetraacetic acid (EDTA), 2% chlorhexidine solution (CHX), etidronic acid (or 1-hydroxyethylidene-1,1-diphosphonic acid, HEDP) and saline solution (SS). The HEDP solution was prepared by adding 1.8 g etidronic acid to 20 mL 2.5 % NaOCl. The concentrations of the individual irrigation solutions were equal in all experimental groups. In five experimental groups and one control group, the samples were exposed to different irrigation protocols for 120 seconds.

The volumes and irrigation times were as follows:
Group 1: H 2.5 ml, 30 s / EDTA 2.5 ml, 60s/ H 2.5 ml, 30 s
Group 2: HEDP 5 ml, 120 s
Group 3: H 2.5 ml, 30 s / EDTA 2.5 ml, 60 s / CHX 2.5 ml, 30 s
Group 4: H 2.5 ml, 30 s / EDTA 2.5 ml, 60 s / SS 2.5 ml, 30 s
Group 5: HEDP 5 ml, 90 s / SS 2.5 ml, 30 s
Negative control group (CG): no irrigation.

After irrigation, the dentin surface was gently dried with air and absorbent paper until no more wetness was visible and bonding was performed immediately. The bonding site was marked with a polymer adhesive strip, which had a hole with a diameter of 2.5 mm and a thickness of 0.2 mm. The GC G-CEM ONE Adhesive Ehancing Primer (AEP, GC, Tokyo, Japan) was applied to the dentin surface according to the manufacturer's instructions (left for 10 s, dried, no polymerization) and composite cement cylinders (GC G-CEM ONE, GC, Tokyo, Japan, LOT 2206131, LOT 2109111) (2.3 mm inner diameter and 3.0 mm height) were created on the bonding surface with a bonding clamp and plastic mold inserts corresponding to UltraTester (Ultradent Products, South Jordan, UT, USA). Bluephase Style LED curing unit (Ivoclar Vivadent, Lichtenstein, Schaan, Switzerland) was used to light-cure the composite cement for 20 s at 1000 mW/cm^2. The samples were left in plastic molds for a further four minutes to complete dark polymerization according to the manufacturer's instructions. The samples were stored in distilled water at 37 °C and 100 % humidity (NUVE ES 120, NÜVE, Ankara, Turkey) for 10 days before shear bond strength testing.

Shear bond strength testing

The ISO 29022 standard was used to test the shear bond strength of composite cements to radicular dentin. The UltraTester (Ultradent Products, SAS Institute Inc., Cary, NC, USA) was used at a constant speed of 1 mm/min until the samples were completely broken.

Irigacijski postupak i adhezijsko cementiranje

Ukupno su pripremljena 72 uzorka koja su nasumično podijeljena u 6 skupina (pet eksperimentalnih i jedna kontrolna skupina) (slika .). Neki uzorci izgubljeni su tijekom procesa lijepljenja jer se utor u kalupu nije savršeno uklopio s rupom u polimernoj ljepljivoj traki, pa je broj uzoraka u različitim skupinama varirao između 8 i 12. Korištene otopine za irigaciju bile su 2,5-postotna otopina natrijeva hipoklorita (H), 17-postotna etilendiamin tetraoctena kiselina (EDTA), 2-postotna otopina klorheksidina (CHX), etidronska kiselina (ili 1-hidroksietiliden-1,1-difosfonska kiselina, HEDP) i fiziološka otopina (SS). Otopina HEDP-a pripremljena je dodavanjem 1,8 g etidronske kiseline u 20 mL 2,5-postotnoga natrijeva hipoklorita. Koncentracije pojedinačnih otopina za irigaciju bile su jednake u svim eksperimentalnim skupinama. U pet eksperimentalnih skupina i jednoj kontrolnoj uzorci su bili izloženi različitim protokolima ispiranja tijekom 120 sekunda.

Količine i vrijeme irigacije bili su sljedeći:
Grupa 1: H 2,5 mL, 30 s / EDTA 2,5 mL, 60 s / H 2,5 mL, 30 s
Grupa 2: HEDP 5 mL, 120 s
Grupa 3: H 2,5 mL, 30 s / EDTA 2,5 mL, 60 s / CHX 2,5 mL, 30 s
Grupa 4: H 2,5 mL, 30 s / EDTA 2,5 mL, 60 s / SS 2,5 mL, 30 s
Grupa 5: HEDP 5 mL, 90 s / SS 2,5 mL, 30 s
Negativna kontrolna skupina (CG): bez irigacije.

Nakon ispiranja površina dentina nježno je osušena zrakom i upijajućim papirom sve dok više nije bilo vidljive vlage te je odmah učinjeno adhezivno cementiranje. Mjesto adhezije označeno je rupom na polimernoj ljepljivoj traki promjera 2,5 mm i debljine 0,2 mm. Na površinu dentina nanesen je primer GC G-CEM ONE (AEP, GC, Tokio, Japan) prema uputama proizvođača (ostavljen 10 s, osušen, bez polimerizacije), a kompozitni cementni cilindri (GC G-CEM ONE, GC, Tokio, Japan, LOT 2206131, LOT 2109111) (unutarnji promjer 2,3 mm i visina 3,0 mm) izrađeni su na spojnoj površini s pomoću stezaljke i plastičnih umetaka s kalupima koji odgovaraju UltraTesteru (Ultradent Products, Južni Jordan, UT, SAD). LED Bluephase polimerizacijska svjetiljka (Ivoclar Vivadent, Lichtenstein, Schaan, Švicarska) korištena je za svjetlosno stvrdnjavanje kompozitnog cementa tijekom 20 s na 1000 mW/cm^2. Uzorci su ostavljeni u plastičnim kalupima još četiri minute da bi se dovršila tamna polimerizacija prema uputama proizvođača. Uzorci su pohranjeni u destiliranoj vodi na 37 °C i 100-postotnoj vlažnosti (NUVE ES 120, NÜVE, Ankara, Turska) tijekom 10 dana prije ispitivanja smične snage veze.

Ispitivanje smične snage sveze

Standard ISO 29022 korišten je za ispitivanje smične snage adhezijske veze kompozitnih cementa na radikularni dentin. UltraTester (Ultradent Products, SAS Institute Inc., Cary, NC, SAD) korišten je konstantnom brzinom od 1 mm/min. do trenutka odvajanja cementa od dentina.

Failure Mode Analysis

After measuring the shear bond strength, the samples were analyzed under a stereomicroscope (Olympus SZX12) to determine the fracture mode. The fractures were categorized as follows: adhesive failure (AD), mixed failure where more than 50 % of the surface is cohesive failure in cement (MFc), mixed failure with more than 50 % of the surface area with adhesive failure (MFa). Cohesive failure in dentin was not observed.

Statistical analysis

The Kolmogorov-Smirnov test was used to test for normal distribution and the Levene test was used to test for homogeneity of variances. A one- way ANOVA was used to assess the differences between the arithmetic means of independent groups. The post-hoc Games-Howell test was used to determine which groups differed significantly. The significance level was set at α=0.05.

Results

Shear bond strength

The highest mean shear bond strength (SBS) was recorded for the control group (M = 22.23 ± 8.38 MPa), while the lowest mean SBS was recorded for H/EDTA/SS (M = 8.8 ± 3.52 MPa) (Table 1). The Kolmogorov-Smirnov normality test showed that the normality assumption was fulfilled at all levels. However, the Levene test (F =3.262, df1=5, df2=52, p<0.05) showed that the variances were heterogeneous. Therefore, a logarithmic data transformation was performed before conducting the ANOVA analysis. After the transformation, the homogeneity of the variances was confirmed by the Levene test (F=1.163, df1=5, df2=52, p=0.340), and the ANOVA was found to be suitable for further analysis. The results of the ANOVA showed a statistically significant difference between the six groups of root dentin samples treated with different irrigation protocols (F=4.138, df1=5, df2=52, p<0.01). The Games-Howell post hoc test was performed to compare all possible combinations of group differences under the assumption that homogeneity of variances was violated (α=0.05). The Games-Howell test identified significant differences between several pairs of groups: CG and H/EDTA/H (p<0.05), CG and H/EDTA/CHX (p<0.05), and CG and H/EDTA/SS (p<0.05). The observed power was 98%. No statistically significant differences were observed between the other groups (Table 2, Figure 2).

Analiza fraktura

Poslije mjerenja smične snage veze uzorci su analizirani pod stereomikroskopom (Olympus SZX12) kako bi se odredio način loma. Lomovi su kategorizirani na sljedeći način: adhezivni lom (AD), mješoviti lom u kojem je više od 50 % površine kohezivni lom u cementu (MFc) i mješoviti lom s više od 50 % površine s adhezivnim lomom (MFa). Kohezivni lom u dentinu nije primijećen.

Statistička analiza

Kolmogorov-Smirnovljev test korišten je za testiranje normalne distribucije, a Leveneov test upotrijebljen je za testiranje homogenosti varijanci. Jednosmjernom ANOVA-om procijenjena je razlika između aritmetičkih sredina nezavisnih grupa. Post-hoc Games-Howellov test korišten je da bi se utvrdilo koje su se skupine značajno razlikovale. Razina značajnosti postavljena je na α = 0,05.

Rezultati

Smična snaga veze

Najveća srednja snaga smične veze (SBS) zabilježena je u kontrolnoj skupini (M = 22,23 ± 8,38 MPa), a najniža srednja vrijednost SBS-a zabilježena za skupinu H/EDTA/SS (M = 8,8 ± 3,52 MPa) (tablica 1.). Kolmogorov-Smirnovljev test normalnosti pokazao je da je pretpostavka normalnosti ispunjena na svim razinama. Međutim, Leveneov test (F = 3,262, df1 = 5, df2 = 52, p < 0,05) pokazao je da su varijance heterogene. Zato je učinjena logaritamska transformacija podataka prije provođenja ANOVA analiza. Nakon transformacije, homogenost varijanci potvrđena je Leveneovim testom (F = 1,163, df1 = 5, df2 = 52, p = 0,340) te je utvrđeno da je ANOVA prikladna za daljnje analize. Rezultati ANOVA-e pokazali su statistički značajnu razliku između šest skupina uzoraka u kojima je korijenski dentin tretiran različitim irigacijskim protokolima (F = 4,138, df1 = 5, df2 = 52, p < 0,01). Games-Howellov post hoc test obavljen je da bi se usporedile sve moguće kombinacije grupne razlike pod pretpostavkom da je povrijeđena homogenost varijanci (α = 0,05). S pomoću Games-Howellova testa identificirane su značajne razlike između nekoliko parova skupina: CG i H/EDTA/H (p < 0,05), CG i H/EDTA/CHX (p < 0,05) te CG i H/EDTA/SS (p < 0,05). Post hoc određena snaga testa bila je 98 %. Nisu uočene statistički značajne razlike između ostalih skupina (tablica 2., slika 2.).

Table 1 Descriptive statistics of the shear bond recordings
Tablica 1. Prikaz deskriptivnih podataka smične snage veze

Group	N	M	SD	SD error
Control group • Kontrolna skupina	8	22,3	8,377	2,962
H/EDTA/ H	12	10,92	5,447	1,572
HEDP	12	14,23	6,057	1,748
H/EDTA/CHX	9	10,82	5,006	1,669
H /EDTA/SS	8	8,80	3,523	1,248
HEDP/SS	9	11,77	10,187	3,396

M - arithmetic mean • aritmetička sredina, SD – standard deviation • standardna devijacija, SD error – standard error • standardna pogreška. EDTA – ethylene-di mine-tetra-acetic acid • etilendiamintetraoctena kiselina, H – sodium hypochlorite • natrijev hipoklorit, SS – saline solution • fiziološka otopina, HEDP – etidronic acid • etidronska kiselina

Table 2 The results of post hoc Games-Howell test. The differences between CG vs. H/EDTA/H, GC vs. H/EDTA/CHX, CG vs. H/EDTA/SS were statistically significant (p<0.05).
Tablica 2. Prikaz rezultata Games-Howellova testa. Razlike izmedu CG-a naspram H/EDTA/H-a, GC naspram H/EDTA/CHX-a, CG naspram H/EDTA/SS-a bile su statistički značajne (p < 0,05).

	Irrigation Protocol • Irigacijski protokol	Difference of arithmetic mean • Razlika aritmetičkih sredina	Standard error • Standardna pogreška	p
Control group • Kontrolna skupina	H/EDTA/H	0.763*	0.218	0.027
	HEDP	0.465	0.201	0.245
	H/EDTA/CHX	0.743	0.218	0.036
	H/EDTA/SS	0.974	0.262	0.026
	HEDP/SS	0.595	0.179	0.052

EDTA – ethylene-di mine-tetra-acetic acid • etilendiamintetraoctena kiselina, H – sodium hypochlorite • natrijev hipoklorit, SS – saline solution • fiziološka otopina, HEDP – etidronic acid • etidronska kiselina

Table 3 Distribution of fracture modes with respect to post space irrigation protocols.
Table 3. Raspodjela vrsta loma s obzirom na protokole irigacije intraradikularnoga prostora

Group • skupina (n)	AD (%)	MFc (%)	MFa (%)
Control group 8 • Kontrolna skupina (8)	5 (62,5)	3 (37,5)	0 (0)
H /EDTA/ H (12)	4 (33,3)	4 (33,3)	4 (33,3)
HEDP (12)	9 (75)	3 (25)	0 (0)
H /EDTA/CHX (9)	5 (55,6)	3 (33,3)	1 (11,1)
H /EDTA/SS (8)	5 (62,5)	2 (25)	1 (12,5)
HEDP/SS (9)	8 (88,9)	1 (11,1)	0 (0)
Total • Ukupno (58)	36 (62,1)	16 (27,6)	6 (10,3)

AD – adhesive failure • adhezivni lom, MFc – mixed failure predominantly cohesive in cement • mješoviti lom pretežno kohezivan u cementu, MFa – mixed failure predominantly adhesive • mješoviti lom pretežno adhezivni.

Failure mode results

Regarding the mode of fracture, adhesive fractures were predominantly observed, followed by mixed adhesive and cohesive fractures in the material and mixed cohesive fractures in the material and adhesive. Cohesive fractures in the dentin were not observed (Table 3, Figure 3).

Discussion

Irrigation solutions used for post-space cleaning and disinfection may affect the structural changes of dentin and surface properties, thus potentially influencing the adhesion of resin cement to the radicular dentin surface (25,34). In previous studies investigating the influence of irrigation solutions on bond strength, dentin was treated with a single irrigation solution in most experimental groups (10, 20, 21, 29, 31, 35). The available literature review shows that the effect of different multi-solution irrigation protocols on the bond strength of G-cem One SARC on radicular dentin primed with AEP has not been investigated. The results of the present study showed that protocols with NaOCl and EDTA resulted in the weakest bond strengths, regardless of the final rinse (NaOCl or SS), and the bond strength was significantly reduced compared to the control group (p<0.05). The null hypothesis which stated that different irrigation protocols would have no influence on bond strength was rejected.

Decalcification route of EDTA

The failure of bonding between SARC and radicular dentin could be attributed to incomplete or insufficient infil-

Analiza fraktura

Tijekom analize fraktura pretežno su uočeni adhezivni lomovi, zatim mješoviti lomovi – dominantno adhezivni i kohezivni u materijalu, te mješoviti lomovi – dominantno kohezivni u materijalu i adhezivni. Nisu primijećeni kohezivni lomovi u dentinu (tablica 3., slika 3.).

Rasprava

Otopine za irigaciju koje se koriste za čišćenje i dezinfekciju intraradikularnoga prostora mogu promijeniti strukturu dentina i svojstva intrakanlane površine utječući na prianjanje kompozitnog cementa na radikularnu površinu dentina (25, 34). U dosadašnjim istraživanjima u kojima su autori ispitivali utjecaj otopina za ispiranje na snagu adhezije, dentin je tretiran jednom otopinom za irigaciju u većini eksperimentalnih skupina (10, 20, 21, 29, 31, 35). Prema dostupnoj literaturi utjecaj različitih irigacijskih protokola s više otopina na snagu veze G-Cem One SARC-a na radikularni dentin tretiran primerom AEP još nije ispitivan. Rezultati ovog istraživanja pokazali su da su protokoli s NaOCl-om i EDTA-om rezultirali najslabijom snagom veze, bez obzira na završno ispiranje (NaOCl ili SS), a snaga veze bila je značajno manja nego u kontrolnoj skupini (p < 0,05). Nulta hipoteza u kojoj se navodi da različiti protokoli irigacije neće utjecati na snagu veze odbačena je.

Dekalcifikacijski učinak EDTA-e

Neuspjeh adhezijskog vezivanja SARC-a na radikularni dentin može se pripisati nepotpunom ili nedovoljnom intration of the material into the partially or fully exposed collagen matrix, depending on the solutions used. This can be attributed to different effects of these solutions on the dentin. EDTA (17%) has a significant etching/demineralization potential, i.e. it follows the decalcification route (36). This means that the salt between the Ca from HAp and the acid is not stable and decalcification progresses, leaving exposed collagen fibers in the dentin (36). Although AEP and SARC contain the acidic functional monomer 10-MDP, which can form stable monomer-Ca salts, much of the mineral content was lost due to the etching effect of EDTA, resulting in reduced formation of stable monomer-Ca salts (36). In this regard, the results of this study are consistent with the findings of Barreto et al. (35), who reported the unsuitability of chelating solutions, including EDTA, for root canal cleaning prior to the application of SARC.

Strategies for improving hybridization of exposed dentin matrix

There are several approaches to mitigate the negative effects of overexposure of the dentin matrix due to EDTA action. These approaches include: the application of CHX, deproteinization by NaOCl and the use of weaker decalcifying agents (such as HEDP). CHX inhibits matrix metalloproteinases, the enzymes the proteolytic action of which can compromise the integrity of the hybrid layer (37). The reported effects of CHX pretreatment of dentin on the longevity of the bond between a fiber post and dentin are controversial, ranging from positive and non-significant to detrimental (28,37,38). In the present study, the application of CHX after EDTA did not lead to a significant improvement in bond strength. However, bond strength was only measured after 10 days, which prevents final conclusions about the long-term effect of CHX application. In addition, the collagen fibrils exposed by the EDTA could be removed by NaOCl, which has a strong proteolytic activity. However, in the present study, neither the application of CHX nor NaOCl after EDTA resulted in improved bond strength. In contrast to the results of this study, Barreto et al (35) reported higher bond strength after application of 2.5% NaOCl for 60 seconds after EDTA. In the present study, NaOCl was applied at the same concentration for only 30 seconds, which may have been a period of time too short to reverse the negative effect of the chelator solution on bond strength. However, scientific confirmation for this and other collagen-depletion strategies to improve hybridization is still lacking. Delgado et al (28) concluded in their systematic review that there is no difference in the bond strength of adhesive materials to dentin when collagen removal is performed after acid etching compared to a conventional hybrid layer. The results of the present study are consistent with the results obtained by Delgado et al.

Weaker chelators preserve HAp on collagen fibers

Another approach to overcome the overexposure of dental fibrils after root canal cleaning is the use of weaker chelators, such as etidronate (HEDP). This allows for more effective material infiltration of the exposed dentin matrix as demineralization is shallower (25). The results of this study

filtracijom materijala u djelomično ili potpuno izloženi kolagenski matriks, ovisno o korištenim tekućinama. To se može pripisati različitim učincima tih otopina na dentin. EDTA (17 %) ima značajan jetkajući/demineralizacijski potencijal, odnosno slijedi dekalcifikacijski put (36). To znači da sol između kalcija iz HAp-a i kiseline nije stabilna i da dekalcifiikacija napreduje, ostavljajući u dentinu izložena kolagena vlakna (36). Iako AEP i SARC sadržavaju kiseli funkcionalni monomer 10-MDP koji može formirati stabilne soli monomera i kalcija, velik dio minerala gubi se zbog dekalcificirajućeg učinka EDTA-e, što rezultira smanjenim stvaranjem stabilnih soli monomera i kalcija (36). Zato su rezultati ove studije u skladu s rezultatima Barreta i suradnika (35) prema kojima kelatori, uključujući i EDTA-u, nisu prikladni za čišćenje intraradikularnog prostora prije primjene SARC-a.

Strategije za poboljšanje hibridizacije eksponiranoga dentinskog matriksa

Nekoliko je pristupa za ublažavanje negativnih učinaka prekomjerne eksponiranosti dentinskoga matriksa u primjeni EDTA-e. Ti pristupi uključuju primjenu CHX-a, deproteinizaciju s pomoću NaOCl-a i uporabu slabijih kelatorskih sredstava za uklanjanje anorganskog sadržaja (kao što je HEDP). CHX inhibira matriksne metaloproteinaze, enzime čije proteolitičko djelovanje može ugroziti integritet hibridnoga sloja (37). Literaturni podatci o učincima predtretmana dentina CHX-om na dugovječnost veze između kompozitne nadogradnje i dentina proturječni su – od pozitivnih i neznačajnih utjecaja do onih štetnih (28, 37, 38). U ovoj studiji primjena CHX-a nakon EDTA-e nije rezultirala značajnim poboljšanjem snage veze. Međutim, snaga veze izmjerena je poslije 10 dana, što ne daje jasan zaključak o dugoročnom učinku primjene CHX-a. Osim toga, izložena kolagenska vlakna nakon EDTA-e mogu se ukloniti NaOCl-om koji ima snažnu proteolitičku aktivnost. Ipak, u ovoj studiji ni primjena CHX-a, ni NaOCl-a nakon EDTA-e nije rezultirala poboljšanom snagom veze. Nasuprot tomu, Barreto i suradnici (35) izvijestili su o većoj snazi veze pri primjeni 2,5-postotnoga NaOCl-a 60 sekunda nakon EDTA-e. U ovom istraživanju NaOCl je primijenjen u istoj koncentraciji samo 30 sekunda, što je možda bilo prekratko da bi se anulirao negativni učinak kelatora na snagu veze. No znanstveni dokazi za takvu i druge tehnike razgradnje dentinskoga matriksa u svrhu poboljšanja hibridizacije još nisu potvrđeni. Delgado i suradnici (28) zaključili su u svojem sustavnom preglednom članku da nema razlike u snazi adhezijske veze između konvencionalnoga hibridnoga sloja u usporedbi s uklanjanjem kolagena poslije jetkanja, a te rezultate podupire i ovo istraživanje.

Slabiji kelatori čuvaju HAp na kolagenim vlaknima

Drugi pristup za sprječavanje prekomjerne eksponiranosti dentinskoga matriksa poslije čišćenja korijenskih kanala jest korištenje slabijih kelatora, kao što je etidronat (HEDP). To omogućuje učinkovitiju infiltraciju materijala u eksponirani dentin kada je demineralizacija plića (25). Rezultati support this approach, as the best results among the HEDP and HEDP/SS groups achieved the best results among the experimental groups. This can be attributed to the weaker decalcification effect of HEDP compared to EDTA and is consistent with the results of Tartari et al. (25) who showed that the mixture of NaOCl and HEDP resulted in more minerals than collagen on the surface of the substrate compared to other decalcifying agents. In addition, HEDP, which belongs to the bisphosphonates, has a high affinity for HAp and increases the surface free energy of dentin when it adsorbs on its surface (30). This increase in surface free energy enables better surface wetting (of low-viscosity materials such as G-Cem One AEP). Surface wetting, together with micromechanical interlocking and chemical bonding, is one of the main mechanisms of adhesion of any dental material (39).

Modified adhesion route of acidic functional monomer-formation of monomer-Ca salts

When the dentin substrate is not treated with chelators, the collagen fibers are not exposed and devoid of hydroxyapatite (HAp), and the SARCs follow a modified adhesion route according to the adhesion/decalcification concept described by Yoshioka et al (36). In such cases, the effect of CHX, NaOCl after EDTA may not be relevant (40). In fact, the results of the present study show better results in the control group in which the dentin was not treated with either disinfectant or chelating solutions. The composition of G-Cem One AEP and the SARC itself theoretically allows the omission of the smear layer removal by chelators. However, such a conclusion should be made with caution. It is important to emphasize that the acidic AEP primer was used in the present study. The low viscosity of the primer can compensate for the low surface free energy of the smear layer-covered dentin, allowing for acceptable wetting. Functional monomers in the primer, such as 10-MDP and 4-MET, can penetrate the smear layer and integrate it into a new interfacial layer (14). In addition, the functional acidic monomers leave only partially demineralized dentin and can form salts with Ca^{2+} released from hydroxyapatite crystals within the thin hybrid layer. Ca-10-MDP salts are resistant to hydrolytic degradation and form a few nanometers thick (4 nm) layer (nano-layering) (39). On the other hand, adequate surface wetting is difficult in the case of SARC due to its viscosity, which is further complicated by the low surface energy of the dentin covered with the smear layer. If the cement is applied without primer, it could theoretically be advantageous to increase the surface free energy by removing the smear layer to improve surface wetting. Furthermore, the removal of the smear layer allows the cement to penetrate into the dentinal tubules, thereby increasing the bond strength (29, 32). This could explain the difference between the results of the present study and the study by Erik et al (32) where irrigation with EDTA prior to cementation with SARC (without previous priming) resulted in the highest bond strength. However, the manufacturer does not recommend a specific irrigation, but advises against the use of EDTA prior to fiber post cementation with G-CEM One. This recommendation is also consistent with the results of the present study.

ove studije potvrđuju takav pristup jer su skupine HEDP i HEDP/SS postigle najbolje rezultate među eksperimentalnim skupinama. To se može pripisati slabijem učinku dekalcifikacije HEDP-a u usporedbi s EDTA-om i u skladu je s rezultatima Tartarija i suradnika (25) koji su pokazali da je mješavina NaOCl-a i HEDP-a rezultirala s više minerala na površini supstrata u usporedbi s drugim dekalcifikacijskim sredstvima. Uz to, HEDP, koji pripada bisfosfonatima, ima visok afinitet za HAp i povećava površinsku slobodnu energiju dentina kada se apsorbira na dentinsku površinu (30). To povećanje površinske slobodne energije omogućuje bolje površinsko prianjanje (materijala niske viskoznosti kao što su G-Cem One AEP). Vlaženje površine, zajedno s mikromehaničkom i kemijskom vezom, jedan je od glavnih mehanizama prianjanja bilo kojega dentalnog materijala (39).

Modificirani put adhezije kiselih funkcionalnih monomera – stvaranje soli monomera i kalcija

Kada se supstrat dentina ne tretira kelatorima, kolagenska vlakna nisu eksponirana i ne manjka hidroksiapatit (HAp), a u skladu s konceptom adhezije/dekalcifikacije koji su opisali Yoshioka i suradnici (36) SARC-i slijede modificirani put adhezije. U takvim slučajevima učinak CHX-a, NaOCl nakon EDTA-e možda nije relevantan (40). Upravo rezultati ove studije pokazuju bolje rezultate u kontrolnoj skupini u kojoj dentin nije tretiran ni dezinficijensom, ni kelatorskim sredstvom. Sastav G-Cem One AEP-a i samoga SARC-a teoretski dopušta izostavljanje uklanjanja zaostatnoga sloja kelatorima. No takvu tvrdnju valja iznijeti s oprezom. Važno je istaknuti da je u ovom istraživanju korišten kiseli primer AEP. Niska viskoznost primera može nadoknaditi nisku površinsku slobodnu energiju dentina prekrivenoga zaostatnim slojem, omogućujući prihvatljivo vlaženje. Funkcionalni monomeri u primeru, kao što su 10-MDP i 4-MET, mogu prodrijeti u zaostatni sloj i integrirati ga u novi međupovršinski sloj (14). Uz to, funkcionalni kiseli monomeri ostavljaju samo djelomično demineralizirani dentin i mogu stvarati soli s Ca^{2+} oslobođenima iz kristala hidroksiapatita unutar tankoga hibridnoga sloja. Soli Ca-10-MDP otporne su na hidrolitičku razgradnju i tvore sloj debljine od nekoliko nanometara (4 nm) (nanoslojevanje) (39). S druge strane, adekvatno površinsko vlaženje teže je u slučaju SARC-a zbog njegove viskoznosti, što se još dodatno komplicira niskom površinskom energijom dentina prekrivenoga zaostatnim slojem. Ako se kompozitni cement nanosi bez primera, teoretski bi moglo biti korisno povećati površinsku energiju uklanjanjem zaostatnoga sloja da bi se poboljšalo površinsko vlaženje. Nadalje, uklanjanjem zaostatnoga sloja omogućuje se cementu da prodre u dentinske tubuluse, čime se povećava snaga veze (29, 32). To bi moglo objasniti razliku između rezultata u ovoj studiji i studije Erika i suradnika (32) u kojoj je irigacija EDTA-om prije cementiranja SARC-om (bez prethodnog stavljanja primera) rezultirala najvećom snagom veze. No proizvođač ne preporučuje konkretnu irigacijsku otopinu, ali savjetuje da se ne upotrebljava EDTA prije cementiranja kompozitne nadogradnje G-CEM One SARC-om. Ta je preporuka također u skladu s rezultatima dobivenima u ovoj studiji.

Failure mode analysis

Adhesive fractures were observed most frequently in all groups, suggesting that the interaction between the luting agent and the dentin and the formation of a hybrid layer is the most common point of failure. Cohesive fractures within the cement were observed more in the groups that were irrigated with NaOCl. This can be explained by the oxidizing effect of NaOCl on polymerization. NaOCl fragments the collagen fibers and chlorinates the end groups, thus producing chloramine-derived radicals. These radicals could potentially interfere with the free radical polymerization of the resin material (in this case cement) at the material-dentin interface (41).

Limitations and future research

One limitation of the present study is the *in vitro* shear bond strength methodology. Although, the push-out test would be more relevant for estimating the bond strength of the post to dentin, the shear bond strength used in this study is clinically relevant as the irrigation solutions affect the dentin. Therefore, the irrigation protocols affect the dentin-cement interface and they do not affect the cement-post interface. In addition, several previous studies addressing the bonding of fiber posts to dentin have shown that failures occur predominantly at the cement-dentin interface, which can be attributed to difficulties in the formation of the hybrid layer (35, 42, 43). In future studies, it would be clinically relevant to analyze the bond strength values after different irrigation protocols of SARCs on radicular dentin with or without the use of AEP prior to cementation. The chemical polymerization initiator in the AEP accelerates the setting of the cement on the dentin surface and contributes to a higher degree of conversion, which may have an impact on long-term bond strength.

Within the limitations of this study, it can be concluded that the application of irrigation solutions immediately prior to adhesive cementation with G-Cem One SARC negatively affects the bond strength to the previously primed radicular dentin. Irrigation with HEDP prior to priming and cementation did not significantly reduce SBS.

Conflict of interest: The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

Ethical approval: The study was approved by the Institutional Ethics Committee of The School of Dental Medicine, University of Zagreb (protocol code 05-PA-30-16-3/2023).

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Authors' Contributions: **M. V., A. I.** – conceived the study, **M. V., D. M.** – performed the investigation and analyzed data; **M. V., A. I.** – wrote the manuscript, **E. K. S., B. J., L. B.** – critically reviewed the manuscript. All authors have read and agreed to the published version of the manuscript.

Analiza fraktura

U svim skupinama najčešći su bili adhezivni lomovi, što upućuje na to da je interakcija između cementa i dentina, tj. stvaranje hibridnoga sloja, najčešća točka neuspjeha. Kohezivni lomovi unutar cementa češće su uočeni u skupinama koje su ispirane NaOCl-om. To se može objasniti oksidacijskim učinkom NaOCl-a na polimerizaciju. NaOCl fragmentira kolagenska vlakna i klorira krajnje ami*no*-skupine, generirajući stvaranje radikala iz kloramina. Ti radikali mogu ometati polimerizaciju slobodnih radikala kompozitnog materijala (u ovom slučaju cementa) na granici materijala i dentina (41).

Ograničenja i buduća istraživanja

Jedno od ograničenja ove studije jest metodologija *in vitro*. Iako bi tzv. test potiska (eng. *push-out* test) bio relevantniji za procjenu snage veze kompozitne nadogradnje na dentin, ispitivanje smične snage veze korišteno u ovoj studiji klinički je relevantno jer irigacijske otopine utječu na dentin. Zato irigacijski protokoli utječu na vezu dentin – cement, a ne na vezu cement – nadogradnja. Osim toga, u nekoliko prethodnih studija koje su se bavile vezom nadogradnje na dentin autori su pokazali da se neuspjeh pojavljuje pretežno na granici cementa i dentina, što se može pripisati poteškoćama u stvaranju hibridnoga sloja (35, 42, 43). U budućim studijama bilo bi klinički relevantno analizirati vrijednosti adhezijske snage poslije upotrebe različitih irigacijskih protokola SARC-a na radikularnom dentinu s AEP-om ili bez upotrebe AEP-a prije cementiranja. Inicijator kemijske polimerizacije u AEP-u ubrzava stvrdnjavanje cementa na površini dentina i pridonosi većem stupnju konverzije, što može utjecati na dugoročnu snagu veze. Unutar ograničenja ove studije može se zaključiti da primjena tekućina za irigaciju, neposredno prije adhezivnog cementiranja G-Cem One SARC-a, negativno utječe na snagu veze s radikularnim dentinom tretiranim primerom. Ispiranje HEDP-om prije postavljanja primera i cementiranja nije značajno smanjilo SBS.

Sukob interesa: Autori nisu bili u sukobu interesa. Osobe koje su financirale istraživanje nisu imale nikakvu ulogu u dizajnu studije, u prikupljanju, analizi ili interpretaciji podataka, te u pisanju rada, ili u odluci o objavi rezultata.

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Doprinos autora: **M. V., A. I.** – osmišljavanje studije; **M. V., D. M.** – istraživanje i analiza podataka; **M. V., A. I.** – pisanje rada; **E. K. S., B. J., L. B.** – kritički pregled rada. Svi su autori pročitali tekst i složili se s objavljenom verzijom rada.

Sažetak
Svrha: Ispitivan je utjecaj različitih irigacijskih protokola na smičnu snagu veze (SBS) samoadherirajućega kompozitnog cementa (SARC) na radikularni dentin prije toga tretiran *primerom*. **Postupci:** Uzorci korijenskog dentina (N = 58) uloženi su u akrilat, ispoliran i nasumično raspoređeni u pet eksperimentalnih skupina (N = 8 – 12) i jednu kontrolnu – CG (N = 8). Korištene irigacijske otopine bile su 2,5-postoni natrijev hipoklorit (H), 17-postotna etilendiamin tetraoctena kiselina (EDTA), 9-postotna etidronska kiselina s hipokloritom (HEDP), 0,9-postotna fiziološka otopina (SS) i 2-postotni klorheksidin (CHX). Svaka eksperimentalna skupina bila je dvije minute podvrgnuta različitom irigacijskom protokolu: 1) H/EDTA/H, 2) HEDP, 3) H/EDTA/CHX, 4) H/EDTA/SS i 5) HEDP/SS. Uzorci u CG-u nisu tretirani nikakvom irigacijskom otopinom. Nakon sušenja naneseni su primer za poboljšanje adhezije veze (AEP) i dvostruko stvrdnjavajući SARC na radikularni dentin s pomoću kalupa koji odgovaraju uređaju Ultra Tester (Ultradent Products, Južni Jordan, SAD). Uzorci su pohranjeni na 37 °C u destiliranoj vodi i poslije 10 dana povrgnuti testiranju snage veze. Rezultati su analizirani s pomoću ANOVA-e i post-hoc Games-Howellova testa, α = 0,05. **Rezultati:** CG je pokazao najviši SBS. Osim HEDP-a i HEDP/SS-a, sve eksperimentalne skupine pokazale su značajno niži SBS u usporedbi s CG-om (p < 0,05). **Zaključci:** Čini se da je HEDP prikladna otopina za čišćenje korijenskog kanala prije cementiranja kompozitnih nadogradnji ojačanih staklenim vlaknima SARC-om uz prethodno nanošenje *primera* na dentin.

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