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Evaluation of the Bond Strength of Bioglass-Based Root Canal Filling Materials

Vrednovanje čvrstoće vezivanja materijala za punjenje korijenskih kanala temeljenih na biostaklu

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Abstract

Objectives: The aim of this study was to evaluate the bond strength of a new endodontic filling material containing added bioactive glass in comparison to a conventional polydimethylsiloxane-based material. **Materials and methods:** Fourteen single-rooted teeth were chemomechanically prepared, divided into two test groups and filled with the test material. The samples were embedded in acrylic resin and sectioned into 1 mm thick slices using a diamond saw. An average of 8 samples was obtained from each tooth (GuttaFlow2 n=37, GuttaFlow bioseal n= 34). The force required to fracture each individual sample was measured using the 'push out' test method. The Kolmogorov-Smirnov test assessed the 'bonding strength' distribution, and the Mann-Whitney U test compared bonding strength differences between the filling material groups. **Results:** The Mann-Whitney U test showed no statistically significant difference between the two tested groups ($P > 0.05$). **Conclusion:** No statistically significant differences in bond strength between tested materials were observed.

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Introduction

The success of endodontic therapy depends on the proper execution of all treatment phases (1). In addition to the mechanical and chemical preparation of root canals, achieving a three-dimensional seal of the treated canal is essential to facilitate the healing of periapical lesions and to prevent reinfection of the endodontic space (2). During the root canal preparation, the goal is to achieve a conical shape (3), as parallel shaping of the canal walls reduces control when placing gutta-percha and sealer (4), which are the most used materials for filling root canals (5). Errors in root canal preparation can result in treatment failure, which is, in case of underfilling due to necrotic and infected tissue that has not been completely removed and causes constant irritation to the periapical tissue (6) or, in case of overfilling, fill-

Uvod

Uspješnost endodontske terapije ovisi o odgovarajućoj provedbi svih faza liječenja (1). Osim mehaničke i kemijske obrade korijenskih kanala, vrlo je važno trodimenzionalno zabrtviti obrađeni korijenski kanal da bi se stvorili uvjeti za uspješno cijeljenje periapikalne lezije i spriječila reinfekcija endodontskog prostora (2). Tijekom obrade korijenskog kanala cilj je postići konični oblik (3) zato što paralelno oblikovanje stijenki kanala smanjuje kontrolu pri postavljanju gutaperke i punila (4), najčešće korištenih materijala za punjenje korijenskih kanala (5). Pogreške u pripremi kanala mogu rezultirati neuspješnim liječenjem, što se u slučaju potpunjenja događa zbog nekrotičnog i inficiranog tkiva koje nije potpuno uklonjeno i uzrokuje konstantnu iritaciju periapikalnog tkiva (6), a u slučaju prepunjenja, materijal za punjenje može

ing material triggering a foreign body reaction (3). Although a wide range of filling materials is available nowadays, none of them is considered ideal (7). According to Grossman, an ideal material should be easy to apply, insensitive to moisture, bactericidal or bacteriostatic, should not cause irritation to periapical tissue and be easily removable if retreatment is required (8 - 10). Increasing attention has been given to materials with bioactive properties (11). The first material of this kind was calcium hydroxide, which stimulates dentine bridge formation (12). Bioactive materials based on calcium silicate cements were later introduced (13). Filling materials incorporating bioactive glass were developed to exploit the advantages of existing materials. Bioactive glass has been used in orthopedics for decades (14). When placed near bone, ions of silicon, phosphate, calcium and sodium are deposited on the surface of bioactive glass, inducing intracellular and extracellular responses that accelerate bone formation (15). Additionally, a silica-rich gel forms on the surface of the material, which reacts with free ions in bodily fluids to form hydroxyapatite. This process creates bonds between the bone, newly formed hydroxyapatite and collagen. Due to its biocompatibility, bioactive glass is considered suitable for use not only in hard dental tissues, but also in soft tissues, such as periapical tissue and dental pulp (14). The primary advantages of these filling materials include biocompatibility, chemical bonding to dentin, accelerated healing of periapical tissue and antimicrobial properties (16).

There is limited literature on the sealing ability of bioactive materials (17). Therefore, this study aimed to evaluate the bond strength of this new polydimethylsiloxane-based material with added bioglass, GuttaFlow bioseal (GFB), to dentin, compared to the bond strength of a standard polydimethylsiloxane-based material, GuttaFlow 2. We used the push-out test, as one of the most reliable and widely used testing methods for simulating clinical conditions (18).

Our hypothesis was that there is no statistical difference between two tested groups, in which case the new material would be considered as good as the standard, GuttaFlow2 material in bond strength.

Materials and methods

The research was approved by the Ethics Committee of the School of Dental Medicine, University of Zagreb, under reference number 05-PA-30-18-5/2023.

The materials included in this study, along with their declared composition are listed in Table 1.

Fourteen intact single-rooted teeth (canines and second premolars) were used for the research. The selected teeth were extracted for medically justified reasons at the Department of Oral Surgery, School of Dental Medicine, University of Zagreb. To prepare the teeth, they were first sterilized and then the soft deposits were removed using curettes. The crowns of the teeth were removed with a water-cooled diamond saw (Isomet 1000, Buehler, Düsseldorf, Germany). The average root length was 16 mm. To determine the working length, a hand K-file #10 (Mani, INC, Utsunomiya, Ja-

izavati reakciju stranog tijela (3). Unatoč širokom rasponu danas dostupnih materijala za punjenje, ni jedan se ne smatra idealnim (7). Prema stajalištu Grossmana, idealni materijal trebao bi biti jednostavan za primjenu, neosjetljiv na vlagu, baktericidan ili bakteriostatski, ne bi trebao izazivati iritaciju periapikalnog tkiva te bi se trebao lako ukloniti u slučaju da je potrebno ponovno liječenje (8 - 10). Sve se veća pozornost posvećuje materijalima s bioaktivnim svojstvima (11). Prvi takav materijal bio je kalcijev hidroksid koji potiče stvaranje dentinskog mosta (12). Bioaktivni materijali na temelju kalcijevih silikatnih cemenata uvedeni su poslije (13). Materijali za punjenje koji sadržavaju bioaktivno staklo razvijeni su da bi se iskoristile prednosti postojećih materijala. Bioaktivno staklo koristi se u ortopediji već desetljećima (14). Kada se postavi u blizini kosti, ioni silicija, fosfata, kalcija i natrija talože se na površini bioaktivnog stakla te potiču unutarstanične i izvanstanične odgovore koji ubrzavaju formiranje kosti (15). Uz to, na površini materijala stvara se gel bogat silicijem koji reagira sa slobodnim ionima u tjelesnim tekućinama tvoreći hidroksiapatit. Taj proces omogućuje stvaranje veza između kosti, novonastalog hidroksiapatita i kolagena. Zbog svoje biokompatibilnosti, bioaktivno staklo smatra se prikladnim ne samo za uporabu u tvrdim zubnim tkivima, nego i u meklima poput periapikalnog tkiva i zubne pulpe (14). Glavne prednosti tih materijala su biokompatibilnost, kemijsko vezanje za dentin, ubrzano cijeljenje periapikalnog tkiva te antimikrobna svojstva (16).

Trenutačno nema dovoljno literature o svojstvu brtvljenja bioaktivnih materijala (17). Zato je cilj ovog istraživanja bio ispitati čvrstoću veze novog materijala na bazi polidimetilsiloksana s dodatkom bioaktivnog stakla, GuttaFlow bioseal (GFB) za dentin, u usporedbi s čvrstoćom veze standardnog materijala na bazi polidimetilsiloksana, GuttaFlow 2. Korišten je test „push-out“, kao jedna od najpouzdanijih i najčešće korištenih metoda ispitivanja za simuliranje kliničkih uvjeta (18).

Naša hipoteza bila je da nema statistički značajne razlike između dviju ispitivanih skupina, u kojem bi slučaju novi materijal u čvrstoći vezivanja bio jednako dobar kao standardni GuttaFlow2.

Materijali i metode

Istraživanje je odobrilo Etičko povjerenstvo Stomatološkog fakulteta Sveučilišta u Zagrebu pod brojem referencije 05-PA-30-18-5/2023.

Materijali uključeni u ovo istraživanje, zajedno s njihovim deklariranim sastavom, navedeni su u tablici 1.

Za istraživanje je korišteno četrnaest netaknutih jednokorijenskih zuba (očnjaci i drugi pretkutnjaci). Odabrani zubi izvađeni su iz opravdanih medicinskih razloga u Zavodu za oralnu kirurgiju Stomatološkog fakulteta Sveučilišta u Zagrebu. Da bi se pripremili, najprije su sterilizirani, a zatim su mekane naslage uklonjene kiretama. Krune zuba uklonjene su dijamantnom pilom (Isomet 1000, Buehler, Düsseldorf, Njemačka), uz vođeno hlađenje. Prosječna duljina korijena bila je 16 mm. Za određivanje radne duljine kroz korijenski kanal proveden je ručni K-file #10 (Mani, INC, Utsu-

Table 1 Declared composition of tested materials (19).**Tablica 1.** Deklarirani sastav testiranih materijala (19)

Material • Materijal	Manufacturer • Proizvođač	Composition • Sastav
GuttaFlow 2	Coltène/Whaledent GmbH Co, KG, Langenau, Germany • Njemačka	Gutta-percha powder, polydimethylsiloxane, zirconium dioxide, platinum catalyst, paraffin oil, silicone oil, micro silver (preservative), pigments • Prah gutaperke, polidimetilsiloksan, cirkonijev dioksid, platinski katalizator, parafinsko ulje, silikonsko ulje, mikro-srebro (konzervansi), pigmenti
GuttaFlow bioseal (GFB)	Coltène/Whaledent GmbH Co, KG, Langenau, Germany • Njemačka	Gutta-percha, polydimethylsiloxane, zinc oxide, bioactive glass-ceramics, barium sulphate, zirconium, platinum catalyst, micro silver, pigments • Gutaperka, polidimetilsiloksan, cinkov oksid, bioaktivna staklokeramika, barijev sulfat, cirkonij, platinski katalizator, mikro-srebro, pigmenti

pan) was passed through the root canal until it was visible on the apex. Subsequently, the instrument was retracted by 0, 5 mm and the obtained length represented the depth of the instrumentation. The roots were instrumented using a device set to a reciprocal motion of 160/30 with a Reciproc R40 instrument (VDW, GmbH, München, Germany). The root canal was irrigated with 1 mL of NaOCl between each instrument insertion, while the final irrigation was performed in the following sequence: 2, 5% NaOCl solution, rinsing with saline, 17% EDTA solution and then saline again. After chemo-mechanical preparation, the teeth were randomly divided into two test groups of seven samples and filled. The first test group was filled with GuttaFlow 2 material, and the second group was filled with GFB. Key properties of GFB include cytocompatibility, low solubility, low porosity and expansion during curing due to its high water absorption capacity (20). Reciproc gutta-percha size #40 was used to fill the root canals of both test groups. The selected material for each experimental group was prepared according to the manufacturer's instructions. The working length for each individual tooth was marked on the corresponding gutta-percha points, which were then dipped into the prepared material and placed into the root canal up to the apical stop. The excess gutta-percha was removed with heated plugger and the gutta-percha was vertically condensed with a cold plugger. The samples were stored in an incubator (ES 120, NUVE, Ankara, Turkey) in test tubes with saline for 30 days at a temperature of 37°C. After the incubation period, the samples were embedded in an acrylic resin (Heraeus Kuzler GmbH, Hanau, Germany). The samples were placed in rubber mold into which the acrylic resin was poured. They were then cut into slices 1 mm thick using a precision diamond saw (Isomet 1000, Buehler, Düsseldorf, Germany) at a speed of 150-200 rotations per minute, with water cooling. The cuts were made perpendicular to the longitudinal axis of the root. The diameters on each side of the sample were measured with a digital calliper (Alpha Tools, Mannheim, Germany). Since the diameters were different on each side of the sample, the bonding surface was calculated from the obtained values using the formula for a truncated cone:

$$\text{Bonding surface} = \pi(R1 + R2)\sqrt{(R1 - R2)^2 + h^2}$$

(Π – constant 3,14; R1 – larger sample diameter; R2 – smaller sample diameter; h – sample thickness).

The bonding surface was measured using a 'push out' test on a universal testing device, the Lloyd device (Model LRX,

nomiya, Japan) dok nije bio vidljiv na apeksu. Zatim je instrument uvučen za 0,5 mm, a dobivena duljina određivala je dubinu instrumentacije. Korijeni su obrađeni uređajem za strojnu obradu korijenskih kanala postavljenim na recipročnu kretnju 160/30 koristeći se Reciproc R40 (VDW, GmbH, München, Njemačka). Korijenski kanal ispran je između svake instrumentacije s 1 mL NaOCl-a, a konačna irigacija provedena je sljedećim redoslijedom: 2,5-postotna otopina NaOCl-a, ispiranje fiziološkom otopinom, 17-postotna otopina EDTA-e i ponovno fiziološka otopina. Poslije kemomehaničke pripreme zubi su nasumično podijeljeni u dvije ispitne skupine sa sedam uzoraka i napunjeni. Prva ispitna skupina punjena je materijalom GuttaFlow 2, a druga GFB-om. Ključna svojstva GFB-a uključuju citokompatibilnost, nisku topivost, nisku poroznost i širenje tijekom stvrdnjavanja zbog visokog svojstva apsorpcije vode (20). Gutaperka Reciproc #40 korištena je za punjenje korijenskih kanala obiju ispitivanih skupina. Odabrani materijali za svaku skupinu pripremljeni su prema uputama proizvođača. Radna duljina za svaki zub označena je na odgovarajućoj gutaperki koja je zatim uronjena u pripremljeni materijal i postavljena u korijenski kanal do apikalnog stopa. Višak gutaperke uklonjen je zagrijanim vertikalnim potiskivačem, a gutaperka je vertikalno kondenzirana hladnim vertikalnim potiskivačem. Uzorci su pohranjeni u inkubator (ES 120, NUVE, Ankara, Turska) u epruvetama s fiziološkom otopinom 30 dana na temperaturi od 37 °C. Nakon inkubacije uronjeni su u akrilatnu smolu (Heraeus Kuzler GmbH, Hanau, Njemačka). Nakon toga postavljeni su u gumeni kalup u koji je ulivena akrilatna smola. Zatim su rezani u slojeve debljine 1 mm preciznom dijamantnom pilom (Isomet 1000, Buehler, Düsseldorf, Njemačka) brzinom od 150 do 200 okretaja u minuti, uz vođeno hlađenje. Rezovi su napravljeni okomito na uzdužnu os korijena. Promjeri na svakoj strani uzorka mjereni su digitalnom pomičnom mjerkom (Alpha Tools, Mannheim, Njemačka). Budući da su promjeri različiti na svakoj strani uzorka, površina veze izračunata je prema dobivenim vrijednostima koristeći se formulom za krnji stožac:

$$\text{Površina vezivanja} = \pi(R1 + R2)\sqrt{(R1 - R2)^2 + h^2}$$

(Π – konstanta 3,14; R1 – veći promjer uzorka; R2 – manji promjer uzorka; h – debljina uzorka).

Površina vezivanja mjerena je testom „push out” na univerzalnom uređaju za testiranje – Lloyd uređaju (Model LRX, Fareham, Engleska). Nastavak od nehrđajućeg čelika promjera 1 mm korišten je za primjenu kompresivnog opte-

Fareham, England). A stainless attachment with a diameter of 1 mm was used to apply compressive load, touching only the filling material. The loading speed was 1 mm/min. The maximum force applied to the cement before fracturing was expressed in Newtons (N). The bonding strength was obtained by dividing the applied force before fracture in N by the previously calculated bonding surface in mm², and it was expressed in Megapascals (MPa).

The distribution of the variable 'bonding strength' was determined using the Kolmogorov-Smirnov test, and the differences in bonding strength between the tested groups of filling materials were examined using the non-parametric Mann-Whitney U test.

Results

The distribution of maximum force and bond strength significantly deviates from a normal distribution (Kolmogorov-Smirnov test, $p < 0.05$). Therefore, the difference between the tested groups was analyzed using the Mann-Whitney U test. On the other hand, the bonding surface area follows a normal distribution.

Figure 1 illustrates the distribution of all samples, showing that for most samples bond strength ranges between 0 and 1. Higher bond strength values are exceptions rather than the rule. Extreme values, defined as those exceeding 2.5 standard deviations, were excluded from the results due to their deviation from the standard distribution. For the first test group (GuttaFlow 2), extreme values were those greater

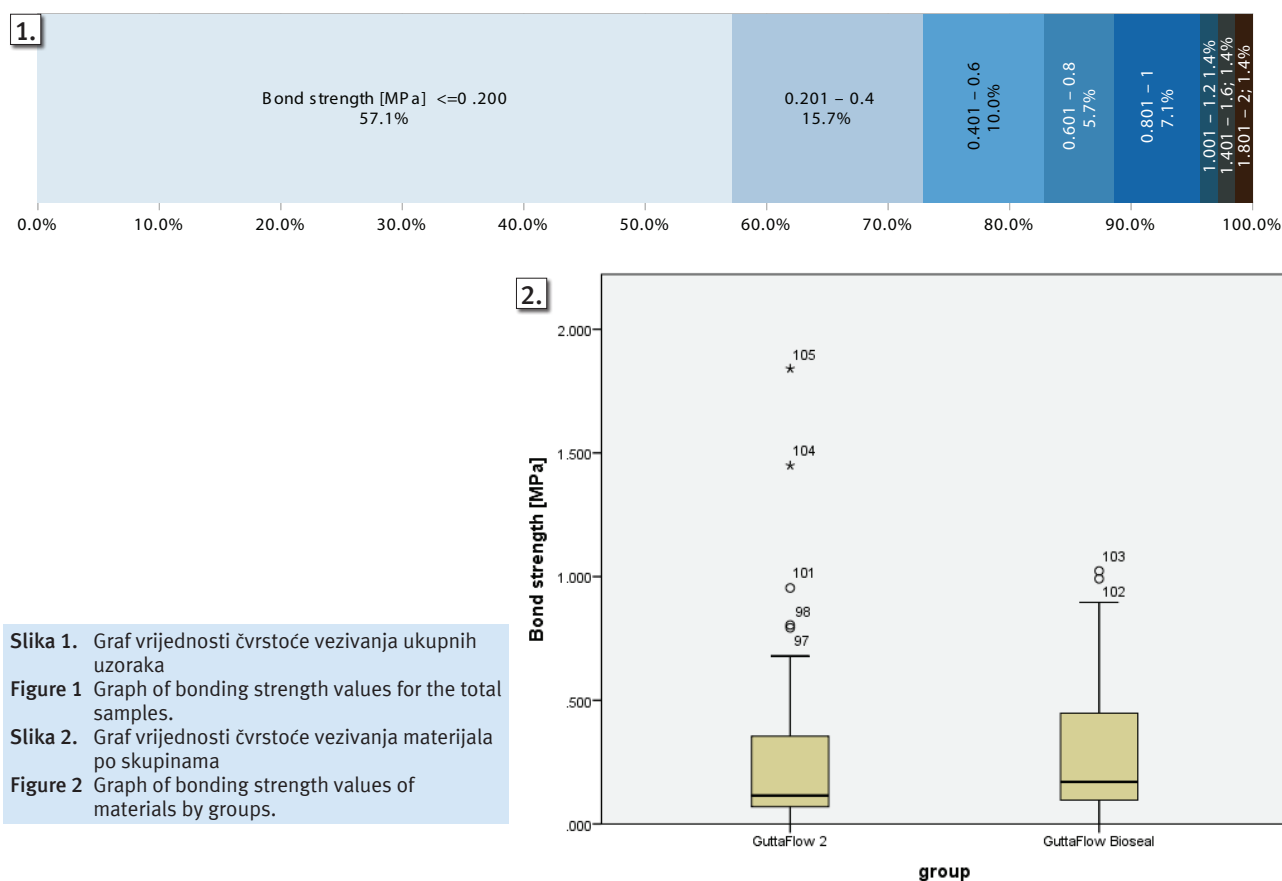
rećenja, dodirujući samo materijal za punjenje. Brzina opterećenja bila je 1 mm/min. Maksimalna sila primijenjena na cement prije puknuća izražena je u njutnima (N). Čvrstoća veze izračunata je dijeljenjem primijenjene sile prije puknuća u N s prethodno izračunatom površinom veze u mm², a izražena je u megapaskalima (MPa).

Distribucija varijable čvrstoće veze određena je Kolmogorov-Smirnovljev testom, a razlike u čvrstoći veze između ispitivanih skupina materijala za punjenje analizirane su neparametrijskim Mann-Whitneyjevim U testom.

Rezultati

Distribucija maksimalne sile i čvrstoće veze značajno odstupa od normalne distribucije (Kolmogorov-Smirnovljev test, $p < 0.05$). Zato su razlike između ispitivanih skupina analizirane Mann-Whitneyjevim U testom. S druge strane, površina veze prati normalnu distribuciju.

Na slici 1. raspodjela je svih uzoraka, pri čemu se za većinu čvrstoća veze kreće između 0 i 1. Veće vrijednosti čvrstoće veze iznimke su, a ne pravilo. Ekstremne vrijednosti, definirane kao one koje prelaze 2,5 standardne devijacije, isključene su iz rezultata zbog odstupanja od standardne distribucije. Za prvu ispitnu skupinu (GuttaFlow 2) ekstremne vrijednosti bile su one veće ili jednake 2, a za drugu ispitnu skupinu (GFB) to su bile vrijednosti veće ili jednake 2,3.



Slika 1. Graf vrijednosti čvrstoće vezivanja ukupnih uzoraka

Figure 1 Graph of bonding strength values for the total samples.

Slika 2. Graf vrijednosti čvrstoće vezivanja materijala po skupinama

Figure 2 Graph of bonding strength values of materials by groups.

Table 2 The values of bonding strength [MPa] bellow certain percentile for GuttaFlow 2 and GuttaFlow Bioseal.**Tablica 2.** Vrijednosti čvrstoće vezivanja [MPa] ispod određenog percentila za GuttaFlow2 i GuttaFlow Bioseal

	Min				1. quartile • 1. kvartil	Median • Medijan	3. quartile • 3. kvartil			Max • Maks.
Percentile	0	5	10	25	50	75	90	95	100	
GuttaFlow2 N = 37	0	.008	.023	.062	.115	.413	.834	1.488		1.84
GuttaFlow bioseal N = 34	0	.032	.053	.092	.173	.556	.944	1.324		2.28

than or equal 2, while for the second test group (GFB), they were values greater than or equal to 2.3.

Figure 2 shows the distribution by groups.

The percentage increase in bond strength is presented in the Table 2. Percentiles indicate the bond strength value below which a specific percentage of the samples is distributed.

Discussion

The results of this study showed no statistically significant differences in the bond strength of GuttaFlow 2 compared to the new root canal filling material, GFB.

The goal is to find materials that achieve excellent sealing by combining bioactive properties with mechanical penetration of the material into dentinal tubules. Both tested materials are based on polydimethylsiloxane (21), with GFB containing bioactive glass particles in addition to powdered gutta-percha, which are responsible for promoting tissue regeneration (22). Bioactive glass also provides antibacterial properties and enhances the biocompatibility of the material in which it is incorporated (23). Both materials have been proven to expand during setting, resulting in better sealing of root canals (17, 24).

Various factors can influence test results, including the sample, substrate, and testing method. Difficulty in obtaining a sufficient number of human teeth often leads to the use of bovine teeth for testing purposes. However, bovine teeth have wider dentinal tubules compared to human teeth, which may affect the bond strength of filling materials (25). For this study, human teeth were used. The results also indicate differences in bond strength between the upper and lower dental arches when tested with shear bond strength methods (26). The condition of the teeth can also impact results. The dentins of intact third molars differ from the sclerotic dentins of teeth with carious and abrasive lesions. Mineral crystals occlude the dentinal tubules of sclerotic dentin, thus reducing bond strength (27). Smaller test samples tend to have higher bond strength (28). It is known that bond strength obtained via shear tests decreases apically (29). Considering chewing forces and temperature fluctuations in the oral environment makes the results more applicable to clinical practice. Temperature changes cause dimensional stress between the tooth and the filling material (30). Previous studies have shown degradation of the bonded interface due to stress from chewing (18). Regarding mechanical testing factors, it has been proven that higher stress at the load application site results in lower bond strength (31). Sood et al. conducted a study (32) demonstrating that variations in load

Na slici 2. raspodjela je po skupinama.

Povećanje čvrstoće vezivanja u postotcima prikazano je u tablici 2. Percentili pokazuju vrijednost čvrstoće vezivanja ispod koje je određeni postotak uzoraka.

Rasprava

Cilj istraživanja bio je pronaći materijale koji postižu izvrsno brtvljenje kombiniranjem bioaktivnih svojstava s mehaničkom penetracijom materijala u dentinske tubuluse. Oba ispitana materijala temelje se na polidimetilsiloksanu (21), s time da GFB, uz usitnjenu gutaperku, sadržava i čestice bioaktivnog stakla koje potiču regeneraciju tkiva (22). Bioaktivno staklo također ima antibakterijska svojstva i povećava biokompatibilnost materijala u koji je inkorporirano (23). Dokazano je da oba materijala ekspanziraju tijekom stvrdnjavanja pa rezultiraju boljim brtvljenjem korijenskih kanala (17, 24).

Na rezultate ispitivanja može utjecati niz čimbenika, uključujući uzorak, supstrat i metodu testiranja. Zbog otežanog prikupljanja dovoljnoga broja ljudskih zuba u svrhu testiranja često se koriste goveđi zubi. Njihovi dentinski tubulusi širi su u usporedbi s ljudskom zubima, što može utjecati na čvrstoću veze punila (25). U ovom istraživanju korišteni su ljudski zubi. Poznate su razlike u čvrstoći veze između gornje i donje čeljusti ispitane testovima smične čvrstoće (26). Stanje zuba također može utjecati na rezultate. Dentin intaktnih trećih molara razlikuje se od sklerotičnog dentina zuba s karijesnim ili abrazivnim lezijama. Mineralni kristali začepljuju dentinske tubuluse u sklerotičnom dentinu, što smanjuje čvrstoću veze (27). Manji uzorci imaju veću čvrstoću veze (28). Također je poznato da vrijednosti čvrstoće padaju apikalno u testovima smične čvrstoće (29). Uzimanje u obzir žvačnih sila i temperaturnih oscilacija u usnoj šupljini čini rezultate primjenjivijima u kliničkoj praksi. Promjene temperature uzrokuju naprezanja između zuba i punila (30). Istraživanja su pokazala degradaciju spoja zbog žvačnih sila (18). Pri testiranju mehaničkih faktora dokazano je da veća naprezanja na mjestu primjene sile rezultiraju nižom čvrstoćom veze (31). Sood i suradnici (32) provedli su istraživanje demonstrirajući da razlike u brzini primjene opterećenja između 0,5 i 10 mm/min ne utječu na vlačnu čvrstoću kompozita. Autori drugog istraživanja istaknuli su manju čvrstoću veze za uzorke opterećene pri brzini od 0,5 i 0,75 mm/min u odnosu na 1 do 1,5 mm/min. (33)

U ovom istraživanju korišten je test „push-out”. Njime se određuje čvrstoća veze između dentina i ispitivanog materi-

application speed between 0.5 and 10 mm/min do not affect the tensile bond strength of composites. Another study showed lower bond strength for samples loaded at 0.5 and 0.75 mm/min compared to those loaded at speeds between 1 and 1.5 mm/min (33).

In this study, the 'push-out' test was used. This test measures the bond strength between dentin and the tested material by recording the resistance of the filling material to force applied to the dentin of the root canal. Tensile force is applied perpendicular to the longitudinal axis of the tooth until the filling material is displaced within the root canal. The 'push-out' test is one of the most reliable and widely used testing methods for simulating clinical conditions (34). However, achieving identical conditions is one of its limitations (35). Many other tests remain in use, but none are ideal. They are divided into micro and macro testing methods. Among macro tests, notable ones include shear bond strength (SBS) tests, tensile bond strength (TBS) tests, and the aforementioned 'push-out' test. Micro tests have the advantage of testing smaller parts of the tooth. However, their benefits are not fully explored (36), or they are unreliable for lower bond strength values (37). The main representatives of this group are micro TBS, micro SBS, and micro 'push-out' tests (36).

In 2024, a study (38) conducted under similar conditions to this one found no statistically significant differences between the tested materials, GuttaFlow 2 and GFB. However, the AH Plus material (Dentsply, DeTrey, Konstanz, Germany) still exhibited the highest bond strength. The thickness of the tested samples in that study was 2 mm, with a loading speed of 0.5 mm/min. On the other hand, results from Dem et al. (34) demonstrated higher bond strength for GFB, despite using the same methodology. These differences could be explained by the incubation method of filled samples and measurements conducted only on slices from the middle third of the root. The samples were stored on moist gauze pre-soaked in phosphate-buffered saline (pH 7.2) for 7 days at 37°C. They were guided by a study according which calcium silicate increases 'push out' properties in such conditions (39). The superior results obtained for AH Plus compared to GFB can be attributed to the formation of covalent bonds with amino groups of collagen in dentin (40). Shenoy et al. (41) demonstrated higher bond strength for GuttaFlow 2 compared to AH Plus, though the difference was not statistically significant. This was attributed to the absence of EDTA solution during the final rinsing of the root canal. EDTA affects the infiltration zone which is crucial for the retention of calcium silicate-based sealers to root canal dentin.

This study contains certain limitations that should be mentioned. During the research, only two materials were tested. We focused on comparing the bond strength of bio-glass-reinforced material with its conventional counterpart to specifically assess the impact of bioglass addition without detracting from the scientific validity or relevance of the findings. The aim of the study was to compare two materials with very similar compositions; however, there are no data on the bonding strength of these materials compared to the best-known root canal sealer, AH Plus. Additionally, the

jala mjerenjem otpornosti punila na silu koja djeluje na dentin korijenskog kanala. Sila se primjenjuje okomito na uzdužnu os zuba, sve dok se punilo ne pomakne u kanalu. Taj je test jedna od najpouzdanijih i najčešće korištenih metoda koja simulira kliničke uvjete (34). Postizanje identičnih uvjeta jedan je od nedostataka toga testa (35). Postoje mnoge druge metode testiranja, ali ni jedna nije idealna. Dije se na makro i mikrotestove. Od makrotestova najvažniji su test smične čvrstoće (engl. *shear bond strength* – *SBS test*), ispitivanje vlačne čvrstoće veze (engl. *tensile bond strength* – *TBS test*) i već spomenuti „push-out”. Mikrotestovi imaju prednost jer se njima ispituju manji dijelovi zuba. Njihova učinkovitost još nije u cijelosti razjašnjena (36) ili su nepouzdani pri niskim vrijednostima čvrstoće veze (37). Među njima su najpoznatiji mikro-TBS, mikro-SBS i mikro „push-out testovi” (36).

Godine 2024. provedeno je istraživanje (38) u uvjetima sličnima ovomu i također nisu pronađene statistički značajne razlike između ispitivanih materijala GuttaFlow 2 i GFB. No materijal AH Plus (Dentsply, DeTrey, Konstanz, Njemačka) i dalje je pokazivao najveću čvrstoću veze. Debljina uzoraka bila je 2 mm, a brzina primjene opterećenja 0,5 mm/min. S druge strane, rezultati istraživanja Dema i suradnika (34) pokazali su veću čvrstoću za GFB, iako je metodologija bila ista. Razlika se može objasniti metodom inkubacije napunjenih uzoraka i činjenicom da su mjerenja provedena samo na srednjoj trećini korijena. Uzorci su čuvani na vlažnoj gazi natopljenom fosfatno puferiranom fiziološkom otopinom (pH 7,2) tijekom 7 dana na 37 °C. Vođeni su studijom prema kojoj kalcijev silikat u tim uvjetima povećava „push-out” svojstva (39). Bolji rezultati za AH Plus u usporedbi s GFB-om pripisuju se formiranju kovalentnih veza s aminoskupinama kolagena u dentinu (40). Shenoy i suradnici (41) pokazali su veću čvrstoću za GuttaFlow 2 u usporedbi s AH Plusom, iako razlika nije bila statistički značajna. To se pripisuje izostanku otopine EDTA-e u završnom ispiranju korijenskog kanala. EDTA utječe na infiltracijsku zonu važnu za retenciju punila temeljenih na kalcijevu silikatu za dentin korijenskog kanala.

Ova studija ima određena ograničenja koja trebaju biti spomenuta. Tijekom istraživanja ispitivana su samo dva materijala. Fokusirali smo se na usporedbu čvrstoće vezivanja materijala ojačanih bioaktivnim staklom s njegovim konvencionalnim ekvivalentom kako bismo procijenili utjecaj bioaktivnog stakla bez umanjivanja znanstvene valjanosti i relevantnosti dobivenih rezultata. Cilj je bio usporediti dva materijala slična sastava, ali ipak nema podataka o usporedbi čvrstoće vezivanja tih materijala u odnosu prema najpoznatijem punilu AH Plus. U studiji je korišteno malo uzoraka, ali veličina uzoraka bila je približno jednaka za obje skupine, dajući dovoljno informacija za svrhe ovoga istraživanja. Treba spomenuti i to da je zabilježen veći broj odstupanja u rezultatima tijekom analize rezultata te širok raspon vrijednosti. Ispitivanje ne daje podatke o utjecaju kemijske obrade korijenskih kanala na čvrstoću veze jer su svi uzorci bili obrađeni jednako. Stoga su rezultati odličan dokaz čvrstoće vezivanja testiranih materijala, ali samo u uvjetima u kojima je istraživanje provedeno. Kako bi rezultati bili primjenjiviji u buduć-

study includes a small number of samples, but the sample size is approximately equal for both tested groups, providing sufficient information for the purposes of this research. On the other hand, it should be noted that the analysis of results detected many outliers and a large span of recorded values. The study does not provide data on the effect of chemical root canal treatment agents on bonding strength, as all samples were treated in the same way. Therefore, the obtained results are excellent evidence of bonding strength of tested materials, but only under the conditions in which the study was conducted. For the result to be more applicable in the future and in clinical practice, further testing is required, taking the mentioned factors into consideration.

Conclusion

The results of this study showed no statistically significant differences between two tested materials. The findings of studies conducted with the same goal vary depending on the preparation and storage of samples and the mechanical characteristics of the testing equipment.

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nosti i u kliničkoj praksi, potrebna su dodatna istraživanja, uzimajući u obzir spomenute čimbenike.

Zaključak

Rezultati ovog istraživanja ne pokazuju statistički značajnu razliku između dvaju ispitivanih materijala. Rezultati istraživanja provedenih s istim ciljem variraju ovisno o pripremi i pohrani uzoraka te mehaničkim svojstvima ispitne opreme.

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Sažetak

Cilj rada: Svrha ovog istraživanja bila je ispitati čvrstoću veze novoga endodontskog punila s dodatkom bioaktivnog stakla u usporedbi s konvencionalnim materijalom na bazi polidimetilsiloksana. **Materijali i metode:** Četrnaest jednokorijenskih zuba kemomehanički je obrađeno, podijeljeno u dvije ispitne skupine te napunjeno ispitivanim materijalima. Uzorci su uloženi u akrilatnu smolu i izrezani dijamantnom pilom u rezove debljine 1 mm. U prosjeku je od svakog zuba dobiveno osam uzoraka (GuttaFlow2 n = 37, GuttaFlow bioseal n = 34). Sila potrebna za frakturu svakog uzorka mjerenja je testom „push-out”. Kolmogorov-Smirnovljev test korišten je za procjenu distribucije čvrstoće veze, a Mann-Whitneyjev U test za usporedbu razlika čvrstoće vezivanja skupina punila. **Rezultati:** Mann-Whitneyjev U test nije pokazao statistički značajnu razliku između dviju ispitivanih skupina (P > 0,05). **Zaključak:** Nije uočena statistički značajna razlika u čvrstoći vezivanja testiranih materijala.

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References

1. Saini HR, Tewari S, Sangwan P, Duhan J, Gupta A. Effect of different apical preparation sizes on outcome of primary endodontic treatment: a randomized controlled trial. *J Endod*. 2012 Oct;38(10):1309–15.
2. Kurt M, Hazar Bodrumlu E. Assessment of root canal filling capacity and quality of the root canal pastes. *Acta Stomatol Croat*. 2025 Mar;59(1):15–25.
3. Johnson WT, Noblett WC. Čišćenje i širenje korijenskog kanala. In: Torabinejad M, Walton RE, editors. *Endodoncija: načela i praksa*. 4th ed. Anić I, translator. Zagreb: Naklada Slap; 2009. p. 258–67.
4. Van der Vyver PJ, Vorster M, Paleker F, de Wet FA. Errors in root canal preparation: a review of the literature and clinical case reports. *S Afr Dent J*. 2019 Jun;74(5):246–54.
5. Hession RW. Long-term evaluation of endodontic treatment: anatomy, instrumentation, obturation—the endodontic practice triad. *Int Endod J*. 1981 Sep;14(3):179–84.
6. Lin LM, Rosenberg PA, Lin J. Do procedural errors cause endodontic treatment failure? *J Am Dent Assoc*. 2005 Feb;136(2):187–93.

7. Glickman GN, Walton RE. Punjenje. In: Torabinejad M, Walton RE, editors. Endodoncija: načela i praksa. 4th ed. Anić I, translator. Zagreb: Naklada Slap; 2009. p. 302.
8. Dobrzańska J, Gołombek K, Dobrzański LB. Polymer material used in endodontic treatment: in vitro testing. Arch Mater Sci Eng. 2012 Dec;58(2):110–5.
9. Katunarić A, Dijanić P, Jurić Kačunić D, Matijević J, Galić N. Efficiency evaluation of various solvents in retreatment of endodontic filling in extracted teeth. Acta Stomatol Croat. 2022 Mar;56(1):2–11.
10. Jurić Kačunić D, Tadin A, Dijanić P, Katunarić A, Matijević J, Trutina-Gavran M, Galić N. Efficacy of reciprocating instruments in retreatment of bioactive and resin-based root canal sealers. Acta Stomatol Croat. 2022 Dec;56(4):338–50.
11. Brzović Rajić V, Mikuš T, Gabrić D, Vuletić M, Verzak Ž. Reparacija i regeneracija koštanog tkiva primjenom bioaktivnog materijala u endodontskoj kirurgiji—prikaz bolesnika. Liječ Vjesn. 2023 Jul 27;145(5–6):216–20.
12. Tiskaya M, Shahid S, Gillam D, Hill R. The use of bioactive glass (BAG) in dental composites: a critical review. Dent Mater. 2021 Feb;37(2):296–310.
13. Baraba A. Materijali za punjenje korijenskih kanala. In: Jukić Krmek S, editor. Pretklinička endodoncija. 1st ed. Zagreb: Medicinska naklada; 2017. p. 122.
14. Hench LL. The story of Bioglass. J Mater Sci Mater Med. 2006 Nov;17(11):967–78.
15. Huang G, Liu SY, Wu JL, Qiu D, Dong YM. A novel bioactive glass-based root canal sealer in endodontics. J Dent Sci. 2022 Jan;17(1):217–24.
16. Camargo RV, Silva-Sousa YTC, Rosa RPF, Mazzichaves JF, Lopes FC, Steier L, et al. Evaluation of the physicochemical properties of silicone- and epoxy resin-based root canal sealers. Braz Oral Res. 2017 Aug 21;31:e72.
17. Lee SH, Oh S, Al-Ghmadi AS, Mandorah AO, Kum KY, Chang SW. Sealing ability of AH Plus and GuttaFlow Bioseal. Bioinorg Chem Appl. 2020 Sep 22;2020:1–8.
18. Arola D. Fatigue testing of biomaterials and their interfaces. Dent Mater. 2017 Apr;33(4):367–81.
19. Saygili G, Saygili S, Tuğlu I, Capar ID. In vitro cytotoxicity of GuttaFlow Bioseal, GuttaFlow 2, AH Plus and MTA Fillapex. Iran Endod J. 2017;12(3):354–9.
20. Gandolfi MG, Siboni F, Prati C. Properties of a novel polysiloxane–guttapercha calcium silicate–bioglass-containing root canal sealer. Dent Mater. 2016 May;32(5):e113–26.
21. Collado-González M, Tomás-Catalá CJ, Oñate-Sánchez RE, Morales JM, Rodríguez-Lozano FJ. Cytotoxicity of GuttaFlow Bioseal, GuttaFlow2, MTA Fillapex, and AH Plus on human periodontal ligament stem cells. J Endod. 2017 May;43(5):816–22.
22. Hench LL, Xynos ID, Polak JM. Bioactive glasses for in situ tissue regeneration. J Biomater Sci Polym Ed. 2004;15(4):543–62.
23. Camargo RV, Silva-Sousa YTC, Rosa RPF, Mazzichaves JF, Lopes FC, Steier L, et al. Evaluation of the physicochemical properties of silicone- and epoxy resin-based root canal sealers. Braz Oral Res. 2017 Aug 21;31:72.
24. Upadhyay ST, Purayil TP, Ballal NV. Evaluation of push-out bond strength of GuttaFlow 2 to root canal dentin treated with different smear layer removal agents. Saudi Endod J. 2018;8(2):128–32.
25. El Mourad AM. Assessment of bonding effectiveness of adhesive materials to tooth structure using bond strength test methods: a review of literature. Open Dent J. 2018 Sep 28;12:664–78.
26. Oztürk B, Malkoç S, Koyutürk AE, Catalbas B, Ozer F. Influence of different tooth types on the bond strength of two orthodontic adhesive systems. Eur J Orthod. 2008 Aug;30(4):407–12.
27. Hevinga MA, Opdam NJ, Frencken JE, Truin GJ, Huysmans MC. Does incomplete caries removal reduce strength of restored teeth? J Dent Res. 2010 Nov;89(11):1270–5.
28. Sano H, Shono T, Sonoda H, Takatsu T, Ciocchi B, Carvalho R, et al. Relationship between surface area for adhesion and tensile bond strength: evaluation of micro-tensile bond test. Dent Mater. 1994 Jul;10(4):236–40.
29. Yuan Y, Shimada Y, Ichinose S, Sadr A, Tagami J. Effects of dentin characteristics on interfacial nanoleakage. J Dent Res. 2007 Oct;86(10):1001–6.
30. Daneshkazemi A, Davari A, Akbari MJ, Davoudi A, Badrian H. Effects of thermal and mechanical load cycling on the dentin micro-tensile bond strength of Single Bond-2. J Int Oral Health. 2015 Aug;7(8):9–13.
31. Furuse AY, Scotti CK, Llerena-Icochea A, Bombonatti JFS, Haragushiku GA, Gonzaga CC. Influence of light activation of simplified adhesives on the shear bond strength of resin cements to a leucite-reinforced ceramic. Eur J Dent. 2018;12(1):3–6.
32. Sood A, Ramarao S, Carounanidy U. Influence of different crosshead speeds on diametral tensile strength of a methacrylate-based resin composite: an in vitro study. J Conserv Dent. 2015;18(3):214–7.
33. Hara AT, Pimenta LA, Rodrigues AL Jr. Influence of cross-head speed on resin-dentin shear bond strength. Dent Mater. 2001 Mar;17(2):165–9.
34. Dem K, Wu Y, Kaminga AC, Dai Z, Cao X, Zhu B. The push out bond strength of polydimethylsiloxane endodontic sealers to dentin. BMC Oral Health. 2019 Aug 8; 19(1):181.
35. Heintze SD, Rousson V, Mahn E. Bond strength tests of dental adhesive systems and their correlation with clinical results- A meta-analysis. Dent Mater. 2015 Apr; 31(4):423–34.
36. El Mourad AM. Assessment of Bonding Effectiveness of Adhesive Materials to Tooth Structure using Bond Strength Test Methods: A Review of Literature. Open Dent J. 2018 Sep 28;12:664–78.
37. Sano H, Chowdhury AFMA, Saikaew P, Matsumoto M, Hoshika S, Yamauti M. The microtensile bond strength test: Its historical background and application to bond testing. Jpn Dent Sci Rev. 2020 Dec; 56(1):24–31.
38. Eissa MH, Boghdadi R, Bedier M. Evaluation of the Push-Out Bond Strength and Penetration of Guttaflow bioseal, Endoseal MTA and Guttaflow 2 versus AH-Plus Root Canal Sealers into the Dentinal Tubules in Mandibular Premolar Teeth (A Randomized in vitro comparative study). J Adv Dent. 2024 Jan; 6(1):181–92.
39. Huffman BP, Mai S, Pinna L, Weller RN, Primus CM, Gutmann JL, et al. Dislocation resistance of ProRoot Endo Sealer, a calcium silicate-based root canal sealer from radicular dentine. Int Endod J. 2009 Jan; 42(1):34–6.
40. Fisher MA, Berzins DW, Bahcall JK. An in vitro comparison of bond strength of various obturation materials to root canal dentin using a push-out test design. J Endod. 2007 Jul; 33(7):856–8.
41. Shenoy N, Shetty K, Jathanna V, Ginjupalli K, Kunaparaju K. Comparative Evaluation of the Push-Out Bond Strength of Three Root Canal Sealers. J Int Dent Med Res. 2020; 13(4):1304–8.