ORIGINAL ARTICLE
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DOI: https://doi.org/10.2298/SGS2303138S
UDC: 616.314-089.48; 616.31:579.84

An influence of finishing procedures and protective coating on the ultrastructure of conventional and hybrid glass ionomer cement restorations

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SUMMARY

Introduction In addition to the advantages of glass ionomer cements that have led to their wide application, hybrid glass ionomer cements have been developed to overcome the shortcomings in mechanical resistance. The aim of the study was to perform an ultrastructural analysis of restorations made from conventional and hybrid glass ionomer cements after recommended finishing procedures and application of a protective coating.

Materials and Methods This study analyzed 30 samples of conventional glass ionomer cement Fuji IX™ and 30 samples of hybrid glass ionomer cement EQUIA Forte HT Fil™. The samples were prepared in cylindrical molds and divided into the three groups. The first group of samples, after adaptation, was left untreated and served as a control group. The second group consisted of samples that were finished with a cylindrical diamond bur with water cooling. The samples in the third group were finished and protected with appropriate coatings (G-COAT PLUS™ and EQUIA Forte Coat™). The samples were analyzed using scanning electron microscopy.

Results The finishing procedures of Fuji IX™ samples significantly reduced crack width ($t = 3.42$, $p < 0.005$; $Z = 3.25$, $p = 0.001$). Similarly, the crack width in EQUIA Forte HT Fil™ samples was also significantly smaller in treated samples ($t = 4.78$, $p < 0.001$; $Z = 4.28$, $p < 0.001$). Ultrastructural analysis of both materials showed the complete absence of cracks in finished samples protected by coatings.

Conclusion Finishing of conventional and hybrid glass ionomer cements results in a reduction in the number of cracks as well as a decrease in their widths, and the protective coatings completely cover remaining cracks.

Keywords: glass ionomer cement; porosity; cracks; ultrastructure; SEM

INTRODUCTION

Numerous positive properties of glass ionomer cements, as well as the constant overcoming of their disadvantages, from their creation in the early 1970s until today, have led to the fact that these materials have become one of the most popular in dentistry [1]. In addition to the most important advantages such as good adhesion to hard dental tissues and fluoride release, the biggest disadvantages of restorations made of these cements are weak mechanical resistance, low wear resistance and high porosity compared to restorations made of composite and amalgam [2]. Porosity is manifested in the presence of voids, pores or cracks inside the material, which results in reduced mechanical properties and increased permeability of material [3].

The need for material based on glass ionomer cement, which would satisfy all patient requirements, contributed to the presentation of the hybrid glass ionomer cement for permanent fillings – EQUIA Forte HT Fil™. The quality of its performance compared to its predecessor EQUIA Forte Fil™ has been confirmed in numerous studies [4]. The manufacturer points out the wide indications of these materials, including class I and II fillings, which are applied in one piece and whose longevity is described by numerous studies [5, 6]. A ten-year follow-up of a patient with EQUIA fillings did not observe a statistically significant difference between these fillings and composite fillings [5]. Due to poor aesthetic properties and possible toxic effects, the suppression of amalgam in many countries has imposed the EQUIA system as a suitable alternative to this mercury alloy, when it comes to class I and II cavities [7]. A study by Moshaverinio et al. [8] indicated that the EQUIA material has higher flexural strength and hardness compared to previous generations of glass ionomers, which makes it a very promising material in restorative dentistry.

Finishing procedures of the material is a treatment during which the surface roughness of the filling and possible irregularities, created during the restoration, are eliminated [9]. The aim of removing these irregularities is to reduce the porosity of the material as well as the accumulation of plaque, which can lead to the colonization of bacteria that has the effect of impairing the aesthetics of the filling itself, but also increasing the chance of tooth decay [10].

Scanning electron microscopy (SEM) is recognized as an extremely effective technique for detailed examination

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Table 1. Classification of tested samples according to the method of preparation
Tabela 1. Klasifikacija uzoraka prema metodi pripreme

Ia group/grupa	Untreated Fuji IX™ samples Netretirani uzorci Fuji IX™	Untreated Fuji IX™ samples Netretirani uzorci Fuji IX™
Ib group/grupa	Untreated EQUIA Forte HT Fil™ samples Netretirani uzorci EQUIA Forte HT Fil™	Untreated EQUIA Forte HT Fil™ samples Netretirani uzorci EQUIA Forte HT Fil™
IIa group/grupa	Fuji IX™ samples – finished with superfine diamond bur with water cooling Fuji IX™ uzorci – ispolirani superfinim dijamantom sa vodenim hlađenjem	Fuji IX™ samples – finished with superfine diamond bur with water cooling Fuji IX™ uzorci – ispolirani superfinim dijamantom sa vodenim hlađenjem
IIb group/grupa	EQUIA Forte HT Fil™ samples – finished with superfine diamond bur with water cooling EQUIA Forte HT Fil™ uzorci – ispolirani superfinim dijamantom sa vodenim hlađenjem	EQUIA Forte HT Fil™ samples – finished with superfine diamond bur with water cooling EQUIA Forte HT Fil™ uzorci – ispolirani superfinim dijamantom sa vodenim hlađenjem
IIIa group/grupa	Fuji IX™ samples – finished and covered with coating G-COAT PLUS™ Fuji IX™ uzorci – ispolirani i premazani G-COAT PLUS™	Fuji IX™ samples – finished and covered with coating G-COAT PLUS™ Fuji IX™ uzorci – ispolirani i premazani G-COAT PLUS™
IIIb group/grupa	EQUIA Forte HT Fil™ samples – finished and covered with coating EQUIA Forte Coat™ EQUIA Forte HT Fil™ uzorci – ispolirani i premazani EQUIA Forte Coat™	EQUIA Forte HT Fil™ samples – finished and covered with coating EQUIA Forte Coat™ EQUIA Forte HT Fil™ uzorci – ispolirani i premazani EQUIA Forte Coat™

of the ultrastructure of materials, due to the possibility of visualizing the microscopic structure of surfaces [11].

The aim of this study was to perform an ultrastructural analysis of the restoration surface of conventional and hybrid glass ionomer cement after recommended finishing procedures and application of protective coatings.

MATERIAL AND METHODS

The research was conducted at the Clinic for Dental Medicine in Niš. The research used a representative of conventional glass cements Fuji IX™ (GC Dental, Tokyo, Japan) and the latest hybrid glass cement EQUIA Forte HT Fil™ (GC Dental, Tokyo, Japan), which were prepared according to the manufacturer's instructions.

Fuji IX™ was manually mixed with a spatula on a paper surface by combining one drop of liquid with one scoop of powder, and then adapted into cylindrical molds measuring 8x3mm using a plastic filling instrument. Individual EQUIA Forte HT Fil™ capsules were mixed for 10 seconds in a mixer (Silamat, Vivadent). After preparation, the material was introduced into cylindrical molds with dimensions of 8x3mm using a capsule applicator and adapted with a placement instrument according to the manufacturer's instructions.

Sixty prepared samples of glass ionomer cements (30 samples of Fuji IX™ and 30 samples of EQUIA Forte HT Fil™) were divided into the three groups. The first group consisted of 20 samples (Ia group – 10 Fuji IX™ samples; Ib group – 10 EQUIA Forte HT Fil™ samples), which were not treated after adaptation and setting. The second group consisted of 20 samples (IIa group – 10 Fuji IX™ samples; IIb group – 10 EQUIA Forte HT Fil™ samples), which, after adaptation and setting, were finished according to the manufacturer's instructions with a superfine cylindrical diamond bur with water cooling. Twenty samples of the third group (IIIa group – 10 Fuji IX™ samples; IIIb group – 10 EQUIA Forte HT Fil™ samples) were, after finishing according to the manufacturer's instructions, protected with a coating that was polymerized with a LED lamp after application. G-COAT PLUS™ coating (GC Dental, Tokyo, Japan) was used to coat Fuji IX™ specimens (IIIa group), while EQUIA Forte Coat™ (GC Dental, Tokyo, Japan) was used to coat EQUIA Forte HT Fil™ specimens (IIIb group) (Table 1).

The preparation of all samples was carried out by one therapist, in order to achieve uniformity in the preparation of samples and the effect of processing. During the treatment, the therapist's movements were even from left to the right. Processing was completed at the moment when the smoothness of the sample was visually assessed by the therapist. After the final processing, the samples were slightly dried for 30 seconds.

Samples were prepared for SEM analysis by first being attached to cylindrical supports with a fixative (Dotite paint xc 12 Carbon JEOL, Tokyo, Japan). In an ion sputtering device (JFC 1100E Ion Sputter JEOL), a thin layer of gold was applied to the samples after vacuuming. For each sample, three micrographs were made at ×500 magnification, on which the crack width was analyzed. ImageJ software was used to estimate the width of the cracks. The distance between the edges of the crack was measured. Thirty distances were measured on each micrograph. Statistical analysis was performed using Mann-Whitney U and Student's t-test in IBM SPSS version 26.0.

RESULTS

Figures 1–3 show micrographs of Fuji IX™ material. Figure 1 shows the ultrastructure of the control sample of group Ia, where rare cracks and pores that dominate the material between the filler particles can be observed. In the samples of group IIa, uniform traces of finishing can be observed over the entire surface and the cracks are significantly reduced. However, pores that penetrate deeper layers of the material are still visible (Figure 2). In Figure 3, which shows the ultrastructure of the sample of group IIIa, a smooth surface covered with a coating can be observed,

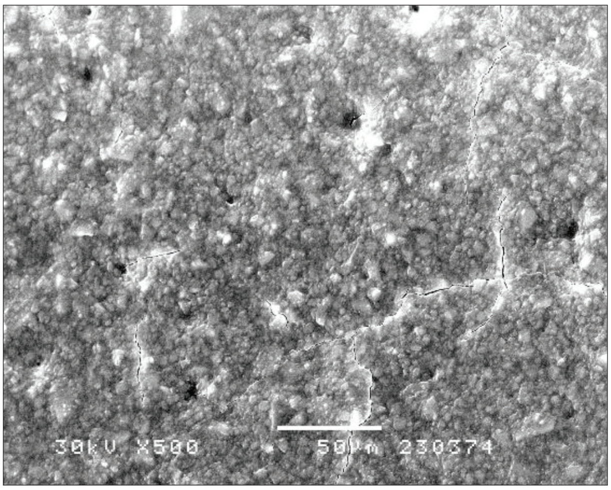

Figure 1. SEM of untreated Fuji IX™ sample
Slika 1. SEM prikaz neobrađenog uzorka Fuji IX™

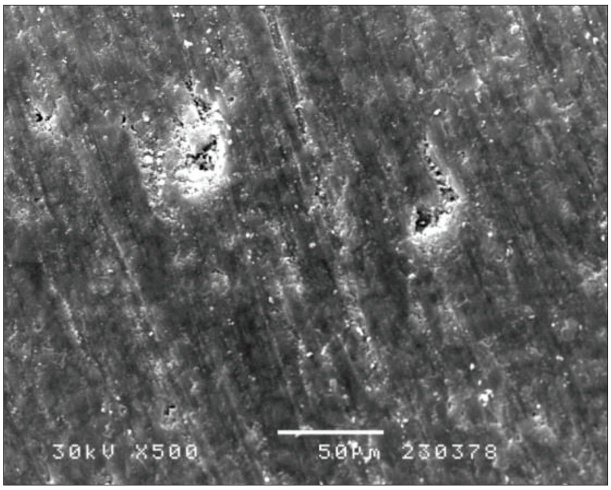

Figure 2. SEM of finished Fuji IX™ sample
Slika 2. SEM prikaz obrađenog uzorka Fuji IX™

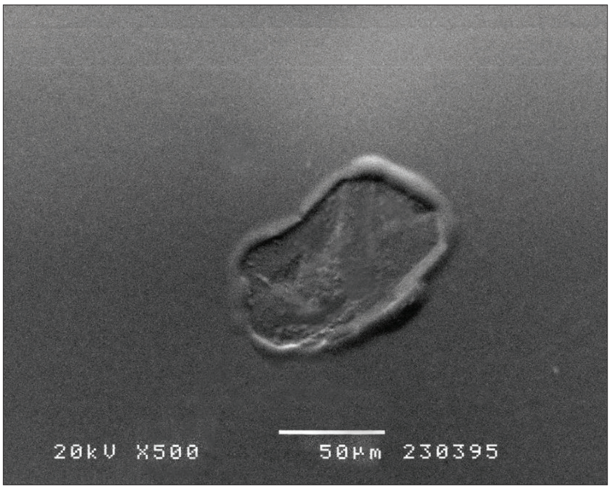

Figure 3. SEM of treated sample of Fuji IX™ with G-COAT PLUS™
Slika 3. SEM prikaz obrađenog uzorka Fuji IX™ premaznog G-COAT PLUS™

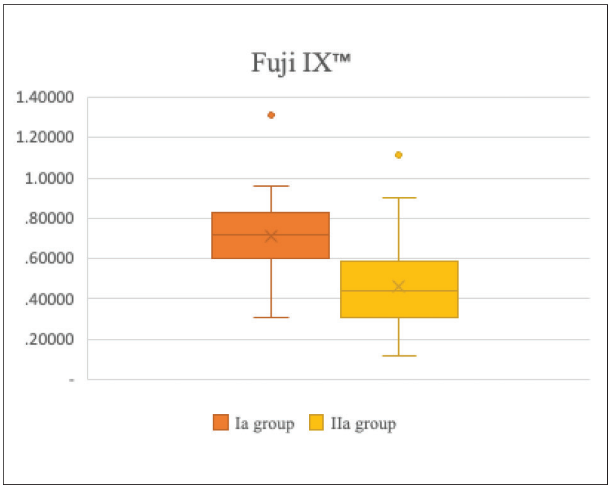

Figure 4. Crack size distribution of untreated and finished Fuji IX™ samples, expressed in µm
Slika 4. Raspodela veličina pukotina na neobrađenim i obrađenim uzorcima Fuji IX™, koje su izražene u µm

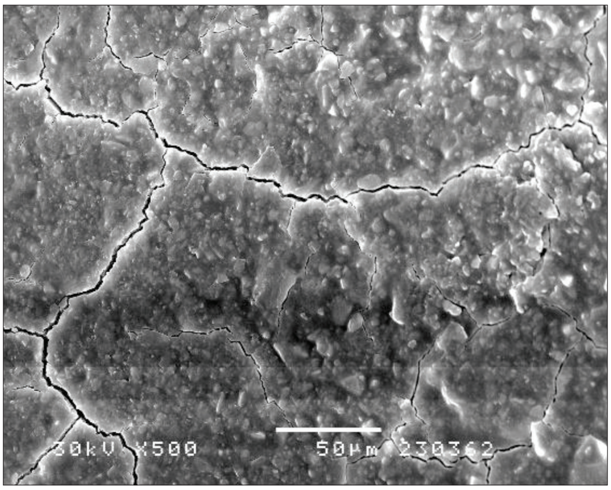

Figure 5. SEM of untreated EQUIA Forte HT Fil™ sample
Slika 5. SEM prikaz neobrađenog uzorka EQUIA Forte HT Fil™

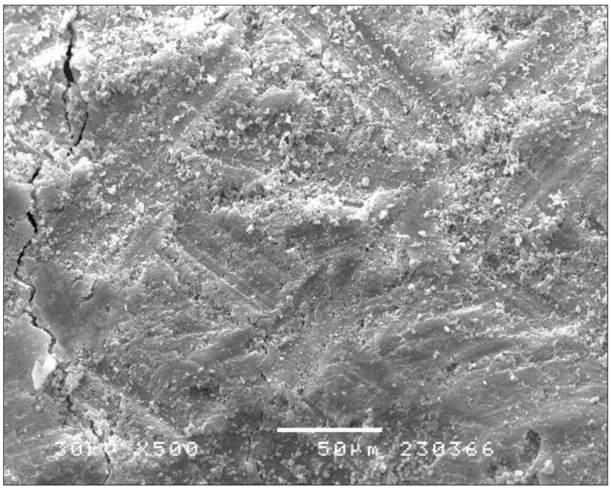

Figure 6. SEM of finished sample of EQUIA Forte HT Fil™
Slika 6. SEM prikaz obrađenog uzorka EQUIA Forte HT Fil™

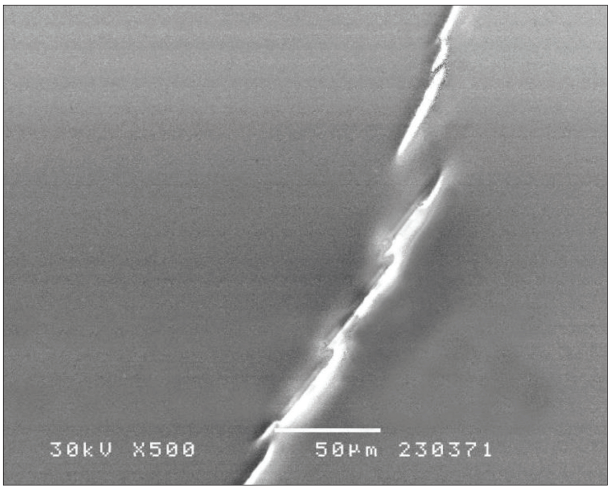

Figure 7. SEM of the treated sample of EQUIA Forte HT Fil™ with EQUIA Forte Coat™
Slika 7. SEM prikaz obrađenog uzorka EQUIA Forte HT Fil™ premaznog EQUIA Forte Coat™

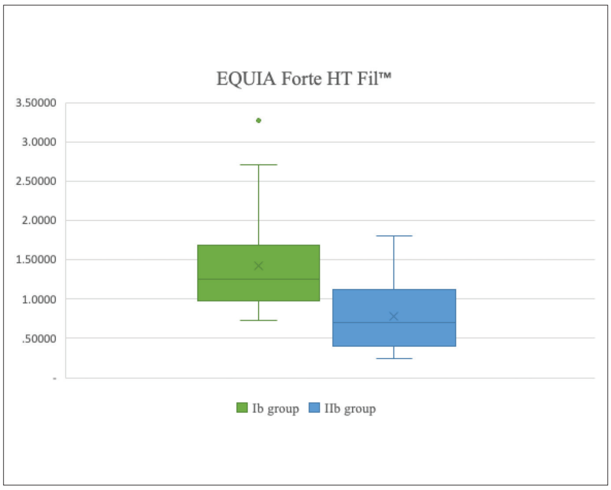

Figure 8. Crack size distribution of untreated and finished EQUIA Forte HT Fil™ samples, expressed in µm
Slika 8. Raspodela veličina pukotina na neobrađenim i obrađenim uzorcima EQUIA Forte HT Fil™, koje su izražene u µm

and rare, clearly delimited places of former pores filled with coat. SEM analysis indicated the reduction of cracks after processing the material, but also the complete filling of pores and cracks after applying the appropriate coating. Figure 4 shows the distribution of crack widths, which were measured on untreated and finished Fuji IX™ samples and expressed in µm. The finishing of the samples resulted in a statistically significant reduction in the width of the cracks ($t = 3.42$, $p < 0.005$; $Z = 3.25$, $p = 0.001$).

Figures 5–7 show micrographs of the EQUIA Forte HT Fil™ material. Figure 5 shows the ultrastructure of the control sample of group Ib, in which a compact surface of the material without pores can be seen, but with the presence of cracks that bypass the filler particles. Figure 6, which presents the ultrastructure of the group IIb sample, shows traces of material finishing and surfaces with rare cracks of smaller width compared to the control samples. On the micrograph of the IIIb group sample, the borders of the cracks are barely visible, and their space is completely filled with coating along its entire length (Figure 7). SEM analysis showed a significant reduction of cracks in the material after finishing and a complete filling of cracks after applying the coating.

Figure 8 shows the distribution of crack widths, which were measured on untreated and finished EQUIA Forte HT Fil™ samples and expressed in µm. The measurements showed that the processing of the samples led to a statistically significant reduction in the crack width ($t = 4.78$, $p < 0.001$; $Z = 4.28$, $p < 0.001$).

DISCUSSION

Thanks to the unique combination of properties, conventional glass ionomer cements are the materials of choice in everyday dental practice. In order to overcome mechanical deficiencies, glass hybrid was developed - innovative materials that combine positive mechanical properties of composites with the fluor protective effect and good adhesion of glass ionomer cement. However, research shows that, dimples and defects can be observed on the surface of these materials after setting, which can negatively affect the clinical properties and durability of the filling [2, 12]. Determining the presence and size of pores in glass ionomer cement-based materials is highly dependent on the type and resolution of the experimental technique used. In the literature, the method of scanning electron microscopy is described for the two-dimensional examination of the surface and the measurement of the width of cracks and pores, while for the three-dimensional examination of porosity, the application of a micro-CT study is more precise [13].

In this study, scanning electron microscopy was used to examine surface defects in materials, which gave a clear insight into the ultrastructure of untreated and treated samples. Analysis of control group samples revealed the presence of microcracks, which is in agreement with numerous studies [8, 14]. Cohesive cracks were observed on samples of both materials, which is in agreement with studies that found cracks in glass ionomer cements occur in the material itself, and not at the junction of the material and the tooth [15]. The appearance of cracks can be explained by the imbalance of water, which occurred in acid-base reactions during cement hardening [16].

The results of this study showed significant occurrence of cracks and pores on the surface of control samples. Data from literature showed that glass ionomer cements proved to be more porous than composites and amalgams [2, 17], so the obtained results were as expected. Porosity testing of EQUIA by Cabello Malagon et al. [18] showed that about 3% of the surface of material was filled with pores. In the mentioned research, it was concluded that porosity of the material was related to the increase in the viscosity of cement. Despite wider cracks on the EQUIA Forte HT Fil™ samples, compared to the Fuji IX™ samples, the absence of pores was also observed in the EQUIA Forte HT Fil™ samples, which is in agreement with the claims from the literature that preparation has the effect on reducing the porosity of cements. Cements prepared in a mixer proved to be less porous than those prepared by hand [19].

Swift et al. [20] pointed out the possibility that the preparation of samples for SEM analysis, may lead to the appearance of additional cracks due to the vacuuming process and the evaporation of water from the cements. However, the same preparation method, which was applied to untreated and finished samples of both groups of materials in this study, did not show a significant effect on the ultrastructure.

The results showed that the recommended finishing procedures with a diamond bur after the cement hardening process resulted in a reduction in the width of microcracks, as well as their removal. Considering that minor irregularities, such as surface protrusions and unevenness [9], are removed during finishing with a diamond bur, it follows that the observed cracks on the untreated samples of EQUIA Forte HT Fil™ and Fuji IX™ were in the surface layers of the material. In the case that the cracks penetrated deeper into the material, diamond bur treatment would not be effective in removing them. The importance of microcracks is reflected in the fact that they can act as stress concentrators that may contribute to material fracture [21]. From this, it follows that stress concentrators, in this case cracks, represent places that should be taken into consideration in order to ensure adequate resistance to stress and to reduce the risk of material cracking. The importance of finishing treatment is also reflected in the reduction of bacterial adhesion to fillings. In an *in vitro* study by Ismail et al. [22], it was determined that resin-modified cements, regardless of the finishing technique, have a smoother surface than conventional glass ionomer cements.

SEM analysis of processed samples with appropriate coatings showed that the cracks were completely filled. This indicates the great importance of the final coating, which can eliminate the problem caused by dehydration during material bonding [23]. The nanofiller coating improves the primary stabilization of the material during curing, but also improves infiltration as well as the closure of surface defects [24]. EQUIA Forte Fil HT™ samples, with a suitable coating, in the study by Brkanović et al. [25] proved to be more resistant to wear compared to those without coating, but no statistical significance was observed. In addition, it has been shown that the appropriate coating affects the reduction of water sorption and the solubility of almost all glass ionomer cement restorations [26]. A study by Ezoji et al. [27] pointed out that treated glass ionomer cement fillings with a suitable coating had significantly less microleakage compared to those without coating protection.

The role of coatings, based on light-curing monomers, that are applied over cement restorations to reduce porosity, can be explained by their property of building a barrier that prevents water exchange during the acid-base reaction of cement hardening. After the subsequent dissolution of the surface layer of the coating after the hardening of the glass ionomer, the cement undergoes secondary maturation under the influence of saliva, and the result is a better restoration [28]. Despite the studies which, in addition to better mechanical properties, highlight the greater resistance of EQUIA Forte HT Fil™ to acid-induced erosions compared to conventional cements such as Fuji IX™ and zinc-reinforced glass ionomer cements such as ChemFil Rock™ [8], the obtained SEM images of this material indicate on the need for additional improvement.

CONCLUSION

The finishing process of the Fuji IX™ and EQUIA Forte HT Fil™ materials leads to reduction in the width of microcracks, as well as to their elimination. The application of appropriate protective coatings affects the filling of the remaining pores and cracks, and obtaining a completely smooth filling surface for both types of cement.

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Received: 10.7.2023 • Accepted: 15.9.2023

Uticaj završne obrade i zaštitnog premaza na ultrastrukturu ispuna od konvencionalnog i hibridnog glasjonomernog cementa

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KRATAK SADRŽAJ

Uvod Pored prednosti i jedinstvenih osobina glasjonomernih cemenata koje su dovele do njihove široke primene, radi prevazilaženja nedostataka u mehaničkoj otpornosti razvijeni su hibridni glasjonomerni cementi.
Cilj ovog rada je bio da se uradi ultrastrukturna analiza površine ispuna od konvencionalnog i hibridnog glasjonomernog cementa nakon preporučene završne obrade i nanošenja zaštitnog premaza.

Materijali i metode U istraživanju je analizirano 30 uzoraka konvencionalnog glasjonomernog cementa Fuji IX™ i 30 uzoraka hibridnog glasjonomernog cementa EQUIA Forte HT Fil™. Uzorci su pripremani u cilindričnim kalupima i podeljeni u tri grupe. Prva grupa uzoraka nakon adaptacije nije obrađivana i služila je kao kontrola. Drugu grupu su činili uzorci koji su nakon adaptacije obrađeni cilindričnim dijamantskim borerom sa vodenim hlađenjem, dok su uzorci treće grupe nakon adaptacije i završne obrade zaštićeni odgovarajućim premazima (G-COAT PLUS™ i EQUIA Forte Coat™). Uzorci su analizirani pomoću skenirajućeg elektronskog mikroskopa.

Rezultati Obrada uzoraka Fuji IX™ je značajno uticala na smanjenje širine pukotina ($t = 3{,}42$, $p < 0{,}005$; $Z = 3{,}25$, $p = 0{,}001$). Širina pukotina kod uzoraka EQUIA Forte HT Fil™ je takođe bila statistički značajno manja kod obrađenih uzoraka ($t = 4{,}78$, $p < 0{,}001$; $Z = 4{,}28$, $p < 0{,}001$). Ultrastrukturna analiza oba materijala je ukazala na potpuno odsustvo pukotina kod obrađenih uzoraka zaštićenih premazima.

Zaključak Završna obrada konvencionalnih i hibridnih glasjonomernih cemenata dovodi do smanjenja broja pukotina, kao i do smanjenja njihovih širina, a zaštitni premazi potpuno prekrivaju preostale pukotine.

Ključne reči: glasjonomerni cementi; poroznost; pukotine; ultrastruktura; SEM

UVOD

Veliki broj pozitivnih osobina glasjonomernih cemenata kao i konstantno prevazilaženje njihovih nedostataka, od svog nastanka početkom 1970-ih pa sve do danas, doveli su do toga da ovi materijali postanu jedni od najpopularnijih u stomatologiji [1]. Pored najvažnijih prednosti kao što su dobra adhezija za tvrda zubna tkiva i otpuštanje fluorida, najveći nedostaci restauracija od ovih cemenata su slaba mehanička otpornost, mala otpornost na trošenje i velika poroznost u poređenju sa restauracijama od kompozita i amalgama [2]. Poroznost se ogleda u prisustvu praznina, pora ili pukotina unutar materijala, što rezultira smanjenjem mehaničkih osobina i povećanom propustljivošću materijala [3].

I pored široke primene konvencionalnih glasjonomernih cemenata, potrebe za materijalom na bazi glasjonomernog cementa, koji bi zadovoljio sve zahteve pacijenata, doprinele su tome da bude predstavljen hibridni glasjonomerni cement za trajne ispune – EQUIA Forte HT Fil™. Kvalitet njegovih performansi u odnosu na prethodnika EQUIA Forte Fil™ potvrđen je u brojnim studijama [4]. Proizvođač ističe široke indikacije ovih materijala, među kojima su i ispuni I i II klase, koji se nanose u jednom komadu i čiju dugovečnost opisuju brojne studije [5, 6]. Desetogodišnje praćenje pacijenta sa ispunima od EQUIA-e nije uočilo statistički značajnu razliku između ovih ispuna i ispuna od kompozita [5]. Zbog loših estetskih osobina i mogućih toksičnih efekata, potiskivanje amalgama u mnogim zemljama nametnulo je EQUIA sistem kao odgovarajuću alternativu ovoj leguri žive, kada su u pitanju kaviteti I i II klase [7]. Studija koju su sproveli Moshaverinia i saradnici [8] ukazuje da materijal EQUIA ima veću čvrstoću na savijanje i veću tvrdoću u poređenju sa prethodnim generacijama glasjonomera, što ga čini veoma perspektivnim materijalom u restaurativnoj stomatologiji.

Završna obrada materijala predstavlja postupak prilikom kog se eliminišu površinska hrapavost ispuna i eventualne nepravilnosti nastale tokom restauracije [9]. Uklanjanje ovih neravnina ima za cilj da smanji poroznost materijala i akumulaciju plaka, koja može dovesti do kolonizacije bakterija, što za posledicu ima narušavanje estetike samog ispuna, ali i povećanje šanse za nastanak karijesa [10].

Skenirajuća elektronska mikroskopija (SEM) priznata je kao izuzetno efikasna tehnika za detaljno ispitivanje ultrastrukture materijala, zbog mogućnosti vizualizacije mikroskopske strukture površina [11].

Cilj ovog rada je bio da se uradi ultrastrukturna analiza površine ispuna od konvencionalnog i hibridnog glasjonomernog cementa nakon preporučene završne obrade i nanošenja zaštitnog premaza.

MATERIJAL I METODE

Istraživanje je obavljeno na Klinici za dentalnu medicinu u Nišu. U istraživanju su korišćeni predstavnik konvencionalnih glasjonomernih cemenata Fuji IX™ (GC Dental, Tokyo, Japan) i najnoviji hibridni glasjonomerni cement EQUIA Forte HT Fil™ (GC Dental, Tokyo, Japan), koji su pripremljeni po uputstvu proizvođača.

Fuji IX™ je ručno zamešan špatulom na papirnoj podlozi sjedinjavanjem jedne kapi tečnosti sa jednom mericom praha, a nakon toga pomoću šestice adaptiran u cilindrične kalupe dimenzija 8 × 3 mm. Pojedinačne kapsule EQUIA Forte HT Fil™ su mešane 10 sekundi u mikseru (Silamat, Vivadent). Nakon pripreme, materijal je pomoću aplikatora za kapsule unesen u cilindrične kalupe dimenzija 8 × 3 mm i adaptiran šesticom i nabijačem po uputstvu proizvođača.

Šezdeset pripremljenih uzoraka glasjonomernih cemenata (30 uzoraka Fuji IX™ i 30 uzoraka EQUIA Forte HT Fil™) podeljeno je u tri grupe. Prvu grupu sačinjavalo je 20 uzoraka (Ia grupa – 10 uzoraka Fuji IX™; Ib grupa – 10 uzoraka EQUIA Forte HT Fil™) koji nakon adaptacije nabijačem i vezivanja nisu obrađivani. Druga grupa se sastojala od 20 uzoraka (IIa grupa – 10 uzoraka Fuji IX™; IIb grupa – 10 uzoraka EQUIA Forte HT Fil™) koji su nakon adaptacije nabijačem i vezivanja obrađivani po uputstvu proizvođača superfinim cilindričnim dijamantskim borerom sa vodenim hlađenjem. Dvadeset uzoraka treće grupe (IIIa grupa – 10 uzoraka Fuji IX™; IIIb grupa – 10 uzoraka EQUIA Forte HT Fil™) nakon adaptacije nabijačem i obrade prema uputstvu proizvođača zaštićeni su premazom koji je nakon aplikovanja polimerizovan LED lampom. Premaz G-COAT PLUS™ (GC Dental, Tokyo, Japan) korišćen je za premazivanje uzoraka Fuji IX™ (IIIa grupa), dok je za premazivanje uzoraka EQUIA Forte HT Fil™ (IIIb grupa) korišćen premaz EQUIA Forte Coat™ (GC Dental, Tokyo, Japan) (Tabela 1).

Pripremu svih uzoraka je realizovao jedan terapeut, kako bi se postigla ujednačenost u pripremi uzoraka i efektu obrade. Prilikom obrade pokreti terapeuta su bili ravnomerni sleva nadesno. Obrada je završena u trenutku kada je terapeut vizuelno procenio glatkoću uzorka. Nakon finalne obrade, uzorci su blago posušeni pusterom u trajanju od 30 sekundi.

Uzorci su pripremljeni za SEM analizu tako što su prvo pričvršćeni za cilindrične nosače uz pomoć sredstva za fiksiranje (Dotite paint xc 12 Carbon JEOL, Tokyo, Japan). U uređaju za jonsko raspršivanje (JFC 1100E Ion Sputter JEOL) na uzorcima je posle vakuumiranja nanesen tanak sloj zlata. Za svaki uzorak su napravljene po tri mikrografije na uvećanju ×500, na kojima je analizirana širina pukotina. Za procenjivanje širine pukotina korišćen je softver ImageJ, uz pomoć kog je mereno rastojanje između ivica pukotine. Na svakoj mikrografiji izmereno je 30 rastojanja. Statistička analiza je izvršena korišćenjem Man-Vitnijevog i Studentovog t-testa u programu IBM SPSS, verzija 26.0.

REZULTATI

Na slikama 1, 2 i 3 prikazane su mikrografije materijala Fuji IX™. Slika 1 prikazuje ultrastrukturu kontrolnog uzorka Ia grupe, na kome se uočavaju retke pukotine i pore koje dominiraju u materijalu između čestica punioca. Na uzorcima IIa grupe zapažaju se ujednačeni tragovi obrade po čitavoj površini, pukotine su značajno smanjene, međutim, i dalje se uočavaju blazne koje zadiru u dublje slojeve materijala (Slika 2). Na Slici 3, koja prikazuje ultrastrukturu uzorka IIIa grupe, uočavaju se glatka površina prekrivena premazom i retka, jasno ograničena mesta nekadašnjih pora ispunjena lakom. SEM analiza je ukazala na smanjenje pukotina posle obrade materijala, ali i na potpuno ispunjavanje pora i pukotina posle nanošenja odgovarajućeg premaza.

Na Slici 4 je prikazana raspodela širina pukotina, koje su izmerene na obrađenim i neobrađenim uzorcima Fuji IX™ i izražene u μm. Obradom uzoraka došlo je do statistički značajnog smanjenja širine pukotina ($t = 3,42$, $p < 0,005$; $Z = 3,25$, $p = 0,001$).

Na slikama 5, 6 i 7 prikazane su mikrografije materijala EQUIA Forte HT Fil™. Slika 5 prikazuje ultrastrukturu kontrolnog uzorka Ib grupe, na kome se vidi kompaktna površina materijala bez pora, ali sa prisutnim pukotinama koje mimoilaze čestice punioca. Na Slici 6, koja prikazuje ultrastrukturu uzorka IIb grupe, vide se tragovi obrade materijala i površina sa retkim pukotinama manje širine u poređenju sa kontrolnim uzorcima. Na mikrografiji uzorka IIIb grupe granice pukotina su slabo vidljive, a njihov prostor je celom dužinom potpuno ispunjen premazom (Slika 7). SEM analiza je pokazala značajno smanjivanje pukotina u materijalu posle obrade i potpuno odsustvo pukotina posle aplikovanja premaza.

Na Slici 8 je prikazana raspodela širina pukotina, koje su izmerene na obrađenim i neobrađenim uzorcima EQUIA Forte HT Fil™ i izražene u μm. Merenja su pokazala da je obrada uzoraka dovela do statistički značajanog smanjenja širine pukotina ($t = 4,78$, $p < 0,001$; $Z = 4,28$, $p < 0,001$).

DISKUSIJA

Zahvaljujući jedinstvenoj kombinaciji osobina, konvencionalni glasjonomerni cementi predstavljaju materijale izbora u svakodnevnoj stomatološkoj praksi. U cilju prevazilaženja mehaničkih nedostataka, razvijeni su glashibridi – inovativni materijali koji objedinjuju pozitivne mehaničke osobine kompozita sa fluoroprotektivnim dejstvom i dobrom adhezijom glasjonomernih cemenata. Međutim, istraživanja pokazuju da se nakon vezivanja na površini ovih materijala mogu uočiti jamice i defekti koji mogu negativno uticati na kliničke osobine i trajnost ispuna [2, 12]. Određivanje prisustva i veličine pora u materijalima na bazi glasjonomernih cemenata u velikoj meri zavisi od tipa i rezolucije eksperimentalne tehnike koja se koristi. U literaturi je za dvodimenzionalno ispitivanje površine i merenje širine pukotina i pora opisana metoda skenirajuće elektronske mikroskopije, dok je za trodimenzionalno ispitivanje poroznosti preciznija primena mikro-CT studije [13].

U ovoj studiji je za ispitivanje površinskih defekata u materijalima korišćena skenirajuća elektronska mikroskopija, koja je dala jasan uvid u ultrastrukturu netretiranih i tretiranih uzoraka. Analizom uzoraka kontrolne grupe uočeno je prisustvo mikropukotina, što je u saglasnosti sa brojnim istraživanjima [8, 14]. Na uzorcima oba materijala uočene su kohezivne pukotine, što je u saglasnosti sa studijama u kojima je zaključeno da se pukotine kod glasjonomernih cemenata događaju u samom materijalu, a ne na spoju materijala i zuba [15]. Pojava pukotina se može objasniti disbalansom vode, do kog dolazi tokom acidobaznih reakcija prilikom stvrdnjavanja cemenata [16].

Rezultati ove studije su pokazali značajnu pojavu pukotina i pora na površini kontrolnih uzoraka. Podaci iz literature govore da su se glasjonomerni cementi pokazali poroznijim od kompozita i amalgama [2, 17], tako da su dobijeni rezultati bili i očekivani. Cabello Malagon i saradnici su ispitivali poroznosti EQUIA-e [18] i pokazali da je oko 3% površine materijala bilo ispunjeno porama. U pomenutom istraživanju je zaključeno da je poroznost materijala povezana sa porastom viskoznosti cemenata. Uprkos širim pukotinama na uzorcima EQUIA Forte HT Fil™, u poređenju sa uzorcima Fuji IX™, u uzorcima EQUIA Forte HT Fil™ je zapaženo i odsustvo pora, što je u saglasnosti sa tvrdnjama iz literature, koje sugerišu da na uticaj na smanjenje poroznosti cemenata ima i način pripreme cemenata. Cementi pripremljeni u mikseru pokazali su se manje poroznijim od onih koji se spremaju ručno [19].

Swift i saradnici [20] ukazali su da priprema uzoraka za SEM analizu, usled procesa vakuumiranja, može da dovede do pojave dodatnih pukotina zbog evaporacije vode iz cemenata, međutim ista metoda pripreme kojoj su bili podvrgnuti i obrađeni i neobrađeni uzorci obe grupe materijala u ovoj studiji nije pokazala značajan uticaj na ultrastrukturu.

Rezultati su pokazali da je preporučena obrada dijamantskim borerom nakon procesa stvrdnjavanja cementa rezultirala smanjenjem širine mikropukotina, kao i njihovim uklanjanjem. S obzirom na to da se prilikom obrade dijamantskim borerom uklanjaju manje nepravilnosti poput površinskih izbočina i neravnina [9], iz ovog proizilazi da su uočene pukotine na neobrađenim uzorcima EQUIA Forte HT Fil™ i Fuji IX™ bile u površinskim slojevima materijala. U slučaju da su pukotine prodirale dublje u materijal, obrada dijamantskim borerom ne bi bila efikasna u njihovom uklanjanju. Značaj mikropukotina ogleda se u tome što one mogu da deluju kao koncentratori naprezanja koji mogu doprineti lomu materijala [21]. Iz ovog proizilazi da koncentratori naprezanja, u ovom slučaju pukotine, predstavljaju mesta koja treba posebno uzeti u razmatranje, kako bi se obezbedila adekvatna otpornost na naprezanje i kako bi se smanjio rizik od pucanja materijala. Značaj završne obrade ogleda se i u smanjenju adhezije bakterija za ispune. U *in vitro* studiji Ismaila i saradnika [22], utvrđeno je da smolom modifikovani cementi, bez obzira na tehniku obrade, imaju glađu površinu od konvencionalnih glasjonomernih cemenata.

SEM analiza obrađenih uzoraka sa odgovarajućim premazima pokazala je kako su pukotine u potpunosti ispunjene. Ovo ukazuje na veliki značaj završnog premaza, kojim se može eliminisati problem izazvan dehidratacijom tokom vezivanja materijala [23]. Premaz od nanopunioca poboljšava primarnu stabilizaciju materijala tokom stvrdnjavanja, ali i infiltraciju i zatvaranje površnih defekata [24]. Uzorci EQUIA Forte Fil HT™, sa odgovarajućim premazom, u studiji Brkanović i saradnika [25] pokazali su se otpornijim na habanje u poređenju sa onima bez premaza, ali nije uočena statistička značajnost. Pored toga, pokazalo se da odgovarajući premaz utiče i na smanjenje sorpcije vode i rastvorljivost gotovo svih restauracija od glasjonomernih cemenata [26]. Studija Ezoji i saradnika [27] istakla je da su obrađeni ispuni glasjonomernih cemenata sa odgovarajućim premazom imali značajno manje mikrocurenje u odnosu na one koji nisu zaštićeni premazom.

Uloga premaza na bazi svetlosnopolimerizujućih monomera koji se aplikuju preko cementa radi prevencije poroznosti može da se objasni njihovom osobinom da grade barijeru koja sprečava razmenu vode tokom acidobazne reakcije stvrdnjavanja cementa. Nakon kasnijeg rastvaranja površnog sloja premaza posle stvrdnjavanja glasjonomera, cement podleže sekundarnoj maturaciji pod dejstvom pljuvačke, a rezultat svega predstavlja kvalitetnija restauracija [28]. Uprkos studijama koje pored boljih mehaničkih osobina ističu i veću otpornost EQUIA Forte HT Fil™ na erozije izazvane kiselinom u poređenju sa konvencionalnim cementima poput Fuji IX™ i cinkom ojačanim glasjonomernim cementima poput ChemFil Rock™ [8], dobijeni SEM prikazi ovog materijala ukazuja na potrebe za dodatnim usavršavanjem.

ZAKLJUČAK

Postupak završne obrade materijala Fuji IX™ i EQUIA Forte HT Fil™ dovodi do smanjenja širine mikropukotina, kao i do njihove eliminacije. Aplikacija odgovarajućih zaštitnih premaza utiče na ispunjavanje preostalih pora i pukotina i dobijanje potpuno glatke površine ispuna kod obe vrste cemenata.