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Evaluation of sorption and solubility of materials based on calcium aluminate

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SUMMARY

Introduction In addition to good biological properties, biomaterials should also possess appropriate physical properties in order to provide stability and longevity at the place of application. The aim of this work was to evaluate physical properties of an experimental nanostructured material based on calcium aluminate (CAL) and calcium silicate (CS).

Material and method The research used nanostructured calcium aluminate synthesized by the hydrothermal sol-gel method from the individual components of calcium aluminate ($CaO{\cdot}Al_2O_3$), calcite ($CaCO_3$) and barium sulfate ($BaSO_4$) as an X-ray contrast agent and calcium silicate (CS). The prepared material was placed in plastic molds with a diameter of 5 ± 0.1 mm and a height of 2 ± 0.1 mm. After setting time, the materials were left in an incubator at 37°C for 24 hours, and then they were removed from the mold and absorption and solubility of the materials was calculated. MTA (Angelus Londrina, Brazil) was used as a control material.

Results The lowest material solubility was recorded with MTA (0.255 mg/mm^3), followed by calcium silicate (0.267 mg/mm^3), and the highest with calcium aluminate (0.725 mg/mm^3). The difference was statistically significant between calcium aluminate and MTA (p = 0.001901) and between calcium aluminate and calcium silicate (p = 0.002550). After 28 days in deionized water, the lowest water sorption was recorded with MTA (0.347 mg/mm^3), followed by calcium silicate (0.357 mg/mm^3), and the highest water sorption was measured with calcium aluminate (0.474 mg/mm^3). Statistically significant differences were observed between calcium aluminate and MTA (p = 0.000283) and between calcium aluminate and calcium silicate (p = 0.001576).

Conclusion Material solubility and water absorption of calcium aluminate-based nanostructured material was significantly higher compared to calcium silicate (CS) and MTA.

Keywords: solubility; water sorption; calcium aluminate; calcium silicate

INTRODUCTION

Mineral trioxide aggregate (MTA) is the material of choice in many indications. It was initially developed as a cement for retrograde root canal filling and for cases of root perforation, but later due to its good clinical properties, it was also used in other indications in endodontics (pulpotomy, direct pulp capping, apexification, for incomplete root growth and for root canal filling [1]. MTA is a biocompatible material and capable of stimulating osteogenesis [2]. It is produced as a powder consisting of fine trioxide particles (tricalcium oxide, silicate oxide and bismuth oxide) and other hydrophilic particles (tricalcium silicate and tricalcium aluminate) responsible for its physical and chemical properties. However, MTA also has some poor properties. Among them, the most significant is the long setting time (which is a consequence of the chemical composition) and difficult clinical manipulation, low resistance to compression, high solubility in a moist environment, the presence and release of arsenic, as well as a high incidence of discoloration of dental structures. A long setting time (more than 3 h) carries the risk of washout immediately after placement, considering that it is placed in cavities that can be contaminated with blood and other tissue fluids [3].

In order to overcome the mentioned disadvantages, new formulations of materials based on calcium silicate and calcium aluminate have been synthesized in recent years [4-5]. Cements based on calcium aluminate, due to the reduced bonding time and associated microstructure, provide great potential in the field of biomaterials. Using nanotechnology, it is possible to overcome certain disadvantages in order to preserve good biological characteristics. Nanotechnology can improve the properties of materials, reduce their mass, increase stability and improve their functionality. One of the main questions that arise in connection with nanomaterials is the safety of their application, their biocompatibility. In recent years at the Institute for Nuclear Research in Vinča

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according to the report of V. Jokanović, a new nanostructured material based on the calcium-aluminate system was synthesized. The material was obtained by hydrothermal sol-gel method and selfexpanding combustion reaction. This mode of synthesis provides high particle activity, faster hydration and short setting time [6-7].

In previous studies *in vivo* conditions, the material based on calcium aluminate has proven biocompatibility and bioinductive potential, the induction of dentine bridge formation and the manifestation of reparative activity [7–12].

In addition to good biological properties, biomaterials should also possess appropriate physical properties to provide stability, longevity at the place of application. In addition to biocompatibility, dimensional stability of endodontic materials is of crucial importance for the success of endodontic treatment. Calcium aluminate-based material binds much faster compared to MTA, has better handling properties and reduced porosity [13, 14].

The aim of this work was to examine physical properties of an experimental nanostructured material based on calcium aluminate (CAL) and calcium silicate (CS).

MATERIAL AND METHOD

The material based on calcium aluminate and calcium silicate was mixed with distilled water in a ratio of 3:1, and the control material (MTA) was prepared according to the manufacturer's instructions. All materials were placed in plastic molds with a diameter of 5 ± 0.1 mm and a height of 2 ± 0.1 mm. For each tested material, 8 samples were made. After setting, the materials were left in an incubator at 37°C for 24 h, then the material samples were taken out of the mold and weighed on a scale (Acculab, Sartorius group, Göttingen, Germany) with an accuracy of 0.0001 g. The obtained material values are marked as initial dry mass (m1). Then the volume (V) of the bonded material samples was calculated based on the volume of the mold in which they were placed. Each sample was separately placed in plastic containers with 5 ml of distilled and demineralized water. Material samples were stored in closed plastic containers and incubated at 37°C for 28 days. After 28 days, the samples were removed from the liquid and the measured mass of the material samples was denoted as (m2). Then the samples were dried with silica gel until a constant mass was established (24 h), which was designated as the final dry mass of the material (m3). For each sample of the tested materials, the values of water sorption and solubility of the material were calculated according to the following formulas:

Water sorption (mg/mm^3) = (m2 - m3) / V

Solubility (mg/mm^3) = (m1-m3) / V [14].

The volume was calculated according to the volume of the mold in which the tested materials were placed.

m1 – initial mass of material samples before immersion in deionized and distilled water

m2 – mass of material samples after 28 days in deionized and distilled water

m3 – mass of material samples after drying

The processing program was used: SPSS 22.0. The obtained results were statistically processed using the t-test of the difference (Student T-test) between the arithmetic means of two small independent samples.

RESULTS

After 28 days in deionized water, the lowest water sorption was found for MTA (0.347 mg/mm^3), followed by calcium silicate (0.357 mg/mm^3), whereas the highest values was observed in calcium aluminate (0.474 mg/mm^3) (Figure 1).

Using the Student's T-test, statistically significant differences were observed between calcium aluminate and MTA (t = -4.913661; p = 0.000283) and between calcium aluminate and calcium silicate (t =-3.908202; p = 0.001576), difference between calcium silicate and MTA was not statistically significant.

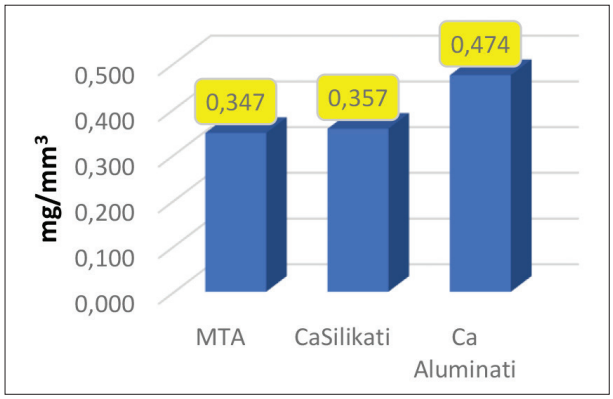

Figure 1. Average water sorption values of the tested materials after 28 days in distilled and deionized water (mg/mm^3)
Slika 1. Prosečne vrednosti sorpcije tečnosti testiranih materijala nakon 28 dana u destilovanoj i dejonizovanoj vodi (mg/mm^3)

The lowest material solubility was found for MTA (0.255 mg/mm^3), followed by calcium silicate (0.267 mg/mm^3). Calcium aluminate had the highest solubility (0.725 mg/mm^3) (Figure 2). Statistically significant difference was found between calcium aluminate and MTA (t= -4.539258; p = 0.001901), as well as between calcium aluminate and calcium silicate (t = -4.318967; p =0, 002550). The difference between MTA and calcium silicate was not statistically significant.

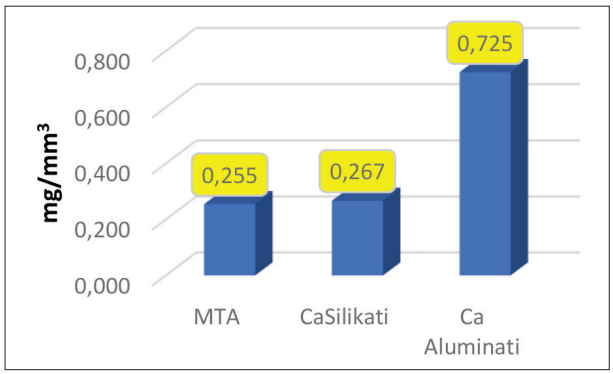

Figure 2. Average solubility values of the tested materials after 28 days in distilled and deionized water (mg/mm^3)
Slika 2. Prosečne vrednosti rastvorljivosti testiranih materijala posle 28 dana u destilovanoj i dejonizovanoj vodi (mg/mm^3)

DISCUSSION

The conditions in which the solubility and porosity of materials (*in vitro*) are evaluated differ in terms of the size of the samples, the amount and type of liquid in which the samples are immersed, the length of the experiment and the type of liquid used [15-16]. In this research, samples of the tested materials were immersed in a liquid (distilled and deionized water) and kept for 28 days, which is in accordance with the methodology of Gandolfi et al, which evaluated the solubility and porosity of the material over a longer period [17, 18].

The water sorption results obtained in this study after 28 days in liquid showed the lowest water sorption for the MTA material. In the case of calcium silicate material, the measured values were slightly higher, while the highest water sorption was observed in calcium aluminate. This result could be explained by the size of the particles in the composition of the material. The nanoparticles in the calcium aluminate formulation are smaller, therefore they are exposed to a larger surface area, which can lead to a more pronounced contact between the particles of the material and the surface on which they react. A larger reactive surface may lead to a consequent higher liquid absorption, that is, a more pronounced contact of the liquid with this material, which results in a higher porosity of the material [19].

The obtained results indicate the hydrophilic nature of all tested cements, considering the fact that high sorption (porosity) of the material was recorded. Porosity is a characteristic of all dental cements that are prepared by mixing powder and liquid and is a consequence of the incorporation of air bubbles during mixing. With calcium aluminate cements, higher values of water absorption were recorded compared to MTA and calcium silicate, which indicates the fact that the material has a highly porous structure.

Solubility and porosity of materials are properties of materials that can directly affect their stability, integrity and durability [20]. It is known that solubility and porosity depends on the ratio of liquid and cement during the preparation [21, 22], on the type of X-ray contrast agent in the composition of the material [23], as well as on the pH value of the environment [24].

Friedland and Rosado claim that all dental cements prepared with water have some degree of porosity due to the incorporation of microscopic air bubbles during the cement mixing process. The same authors claim that the presence of amorphous pores and capillary structure in the composition of MTA is another important cause of the porosity of this material [21].

Luz et al. claim that the porosity of calcium aluminate cement decreases over time, which may be related to the continuity of formation and the deposition of hydrate phases in the newly created pores [25, 26].

Ivone Regina de Oliveira et al. claim that the key to the physical properties is the X-ray contrast medium. The mentioned authors suggest the addition of 15%ZnO:10%Bi_2O_3 in the composition of calcium aluminate as the most suitable agent that can achieve the best compromise between good physical and mechanical properties and X-ray contrast. They indicate that the addition of 15%ZnO:10%Bi_2O_3 results in a decrease in porosity [27, 28, 29]. The same authors claim that Bi_2O_3 increases the porosity and reduces the mechanical strength of calcium aluminate cement whose particles are of different sizes and elongated shapes.

Garcia et al. evaluated solubility and porosity of materials based on calcium aluminate EB (EndoBinder) with three different radiopacifiers agents: bismuth oxide ($B_{i2}O_3$), zinc oxide (ZnO) and zirconium oxide (ZrO_2). EndoBinder showed similar behavior to MTA, regardless of X-ray contrast. The authors claim that the long time of MTA binding leads to its instability, to greater solubility and disintegration, and as a result there is a greater release of components present in the cement itself. The solubility of MTA was 5.74% for gray and 6.65% for white, and these values are above the limit values of (3%) proposed by the specifications [13].

Parreira et al. in their study (2016) claim that the addition of ZnO, as well as hydroxyapatite in calcium aluminate cement formulations, results in a reduction in the porosity level of the tested samples and a reduction in pores after contact with simulated body fluids. ZnO is a biomaterial capable of inducing mineralization processes [30].

However, some authors think that the high solubility of the material can be a benefit, from a biological and physico-chemical point of view, because more calcium ions are released into the surrounding tissue, just as a high pH can cause a greater antibacterial effect [30]. Therefore, applied *in vivo* conditions, these cements become a source of calcium and hydroxyl ions with consequent bioactivity and antimicrobial effect.

Studies have shown that calcium ions are the main component detected in solubility, showing that cement solubility is an important phenomenon in the release of calcium and hydroxyl ions into the periodontal tissue, which can affect reparative processes [31].

However, it should be mentioned that in clinical conditions only a small part of the material comes into contact with tissue fluids, compared to laboratory tests where the entire material sample is immersed in liquid, therefore the osmotic effect is more pronounced.

It must be considered that the measurement of differences in the weight of cement samples may also record decay processes that may not be the result of dissolution. For example, material particles may fall out of the cement structure during storage of the material in liquid over time [32].

CONCLUSION

The solubility and water absorption of the experimental nanostructured material based on calcium aluminate was significantly higher compared to calcium silicates and MTA.

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Ispitivanje sorpcije i rastvorljivosti materijala na bazi kalcijum-aluminata

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KRATAK SADRŽAJ

Uvod Pored dobrih bioloških osobina, biomaterijali treba da poseduju i odgovarajuća fizička svojstva kako bi obezbedili stabilnost, odnosno dugotrajnost na mestu aplikacije.

Cilj ovog rada je bio da se ispitaju fizička svojstva eksperimentalnog nanostrukturnog materijala na bazi kalcijum-aluminata i kalcijum-silikata.

Materijal i metod rada U istraživanju je korišćen nanostrukturni kalcijum-aluminat sintetisan hidrotermalnom sol-gel metodom od pojedinačnih komponenata kalcijum-aluminata ($CaO{\cdot}Al_2O_3$), kalcita ($CaCO_3$) i barijum-sulfata ($BaSO_4$) kao rendgenskog kontrastnog sredstva i kalcijum-silikata. Zamešani materijal je postavljen u plastične kalupe prečnika 5 ± 0,1 mm i visine 2 ± 0,1 mm. Nakon vezivanja materijali su ostavljeni u inkubatoru na 37°C tokom 24 h, a potom su izvađeni iz kalupa i proverene su apsorpcija i rastvorljivost materijala. Kao kontrolni materijal korišćen je MTA (Angelus Londrina, Brazil).

Rezultati Najmanja rastvorljivost materijala zabeležena je kod MTA (0,255 mg/mm³), zatim kod kalcijum-silikata (0,267 mg/mm³), a najveća kod kalcijum-aluminata (0,725 mg/mm³). Razlika je bila statistički značajna između kalcijum-aluminata i MTA (p = 0,001901) i između kalcijum-aluminata i kalcijum-silikata (p = 0,002550). Nakon 28 dana u dejonizujućoj vodi, najmanja sorpcija tečnosti zabeležena je kod MTA (0,347 mg/mm³), potom kod kalcijum-silikata (0,357 mg/mm³), a najveća sorpcija tečnosti izmerena je kod kalcijum-aluminata (0,474 mg/mm³). Statistički značajne razlike uočene su između kalcijum-aluminata i MTA (p = 0,000283) i između kalcijum-aluminata i kalcijum-silikata (p = 0,001576).

Zaključak Rastvorljivost materijala i apsorpcija tečnosti kod nanostrukturnog materijala na bazi kalcijum-aluminata je bila značajno veća u poređenju sa kalcijum-silikatom i MTA.

Ključne reči: rastvorljivost; apsorpcija; kalcijum-aluminat; kalcijum-silikat

UVOD

Mineralni trioksidni agregat (MTA) danas je materijal izbora u brojim indikacijama. U početku je razvijen kao cement za retrogradno punjenje kanala i za slučajeve kod perforacije korena zuba, ali je kasnije zbog svojih dobrih kliničkih osobina primenjivan i u drugim indikacijama u endodonciji (pulpotomija, direktno prekrivanje pulpe, apeksifikacije, kod nezavršenog rasta korena i za punjenje kanala korena zuba) [1]. MTA je biokompatibilan materijal i sposoban da stimuliše osteogenezu [2]. Proizvodi se kao prah koji se sastoji od finih čestica trioksida (trikalcijev oksid, silikatni oksid i bizmutov oksid), te ostalih hidrofilnih čestica (trikalcijev silikat i trikalcijev aluminat) odgovornih za njegova fizička i hemijska svojstva. Međutim, MTA poseduje i neke loše osobine. Među njima su najznačajnije dugo vreme stvrdnjavanja (koje je posledica hemijskog sastava) i otežana klinička manipulacija, odnosno niska otpornost na kompresiju, visoka rastvorljivost u vlažnoj sredini, prisustvo i oslobađanje arsena, kao i visoka incidenca prebojavanja dentalnih struktura. Dugo vreme vezivanja (više od 3 h) nosi rizik od ispiranja materijala sa mesta aplikacije s obzirom na to da se postavlja u kavitete koji mogu biti kontaminirani krvlju i drugim tkivnim fluidima [3].

U cilju prevazilaženja navedenih problema, poslednjih godina sintetisane su brojne nove formulacije materijala na bazi kalcijum-silikata (KS) i kalcijum-aluminata (KAL) [4]. Cementi na bazi KAL zbog smanjenog vremena vezivanja i povezane mikrostrukture obezbeđuju veliki potencijal na polju biomaterijala. Upotrebom nanotehnologije moguće je prevazići pomenute nedostatke prilikom sintetisanja materijala u cilju očuvanja dobrih bioloških karakteristika. Nanotehnologijom se mogu poboljšati osobine materijala, smanjiti njihova masa, povećati stabilnost i unaprediti njihova funkcionalnost. Jedno od glavnih pitanja koje se postavlja u vezi sa nanomaterijalima je bezbednost njihove primene, tj. njihova biokompatibilnost. Poslednjih godina na Institutu za nuklearna istraživanja u Vinči, prema izveštaju V. Jokanovića, sintetisan je novi nanostrukturni materijal na bazi KAL sistema dobijen hidrotermalnom sol-gel metodom i samoširećom reakcijom sagorevanja. Ovakav način sinteze obezbeđuje visoku aktivnost čestica, bržu hidrataciju i kratko vreme vezivanja [6].

U dosadašnjim ispitivanjima u *in vivo* uslovima materijal na bazi KAL je pokazao biokompatibilnost i bioinduktivni potencijal, tj. indukciju stvaranja dentinskog mosta i ispoljavanje reparatorne aktivnosti [8–12].

Pored dobrih bioloških osobina, biomaterijali treba da poseduju i odgovarajuća fizička svojstva kako bi obezbedili stabilnost, odnosno dugotrajnost na mestu aplikacije. Pored biokompatibilnosti, dimenzionalna stabilnost endodontskih materijala od krucijalnog je značaja za uspeh endodontskog tretmana. Materijal na bazi KAL vezuje se dosta brže u poređenju sa MTA, poseduje bolja svojstva rukovanja i redukovanu poroznost [13, 14].

Cilj ovog rada je da se ispitaju fizička svojstva eksperimentalnog nanostrukturnog materijala na bazi KAL i KS.

MATERIJAL I METOD RADA

Materijal na bazi KAL i KS su zamešani sa destilovanom vodom u odnosu 3 : 1, a kontrolni materijal (MTA) zamešan je prema

uputstvu proizvođača. Svi materijali su postavljeni u plastične kalupe prečnika 5 ± 0,1 mm i visine 2 ± 0,1 mm. Za svaki testirani materijal napravljeno je po osam uzoraka. Nakon vezivanja materijali su ostavljeni u inkubatoru na 37°C tokom 24 h, potom su uzorci materijala izvađeni iz kalupa i izmereni na vagici (Acculab, Sartorius group, Getingen, Germany) sa preciznošću od 0,0001 g. Dobijene vrednosti materijala označene su kao inicijalna suva masa (m1). Zatim je izračunata zapremina (V) uzoraka vezanih materijala na osnovu zapremine kalupa (valjka) u kome su postavljeni. Svaki uzorak posebno je postavljen u plastične kontejnere sa 5 ml destilovane i demineralizovne vode. Uzorci materijala su čuvani u zatvorenim plastičnim kontejnerima i inkubirani na 37°C tokom 28 dana. Posle 28 dana uzorci su izvađeni iz tečnosti i izmerena masa uzoraka materijala označena je kao m2. Zatim su uzorci isušeni gelom silika do uspostavljanja konstantne mase (24 h), koja je označena kao finalna suva masa materijala (m3). Potom su za svaki uzorak testiranih materijala izračunate vrednosti sorpcije tečnosti i rastvorljivosti materijala prema sledećim formulama:

Sorpcija tečnosti (mg/mm^3) = (m2 - m3) / V

Rastvorljivost (mg/mm^3) = (m1 - m3) / V [14].

Volumen je izračunat prema zapremini kalupa u kome su postavljeni testirani materijali.

m1 – inicijalna masa uzoraka materijala pre potapanja u dejonizujuću i destilovanu vodu

m2 – masa uzoraka materijala posle 28 dana u dejonizujućoj i destilovanoj vodi

m3 – masa uzoraka materijala nakon isušivanja

Korišćen je program za obradu SPSS 22.0 Dobijeni rezultati su statistički obrađeni primenom t-testa razlike (Studentov t-test) između aritmetičkih sredina dva mala nezavisna uzorka.

REZULTATI

Posle 28 dana u dejonizujućoj vodi, najmanja sorpcija tečnosti zabeležena je kod MTA (0,347 mg/mm^3), potom kod KS (0,357 mg/mm^3), dok je najveća sorpcija tečnosti izmerena kod KAL (0,474 mg/mm^3) (Grafikon 1).

Primenom Studentovog t-testa uočene su statistički značajne razlike između KAL i MTA (t = -4,913661; p = 0,000283) i između KAL i KS (t = -3,908202; p = 0,001576), dok razlika između KS i MTA nije bila statistički značajna.

Najmanja rastvorljivost materijala zabeležena je kod MTA (0,255 mg/mm^3), zatim kod KS (0,267 mg/mm^3). Kod KAL je uočena najveća rastvorljivost (0,725 mg/mm^3) (Grafikon 2). Utvrđena je statistički značajna razlika između KAL i MTA (t = -4,539258; p = 0,001901), kao i između KAL i KS (t = -4,318967; p = 0,002550). Razlika između MTA i KS nije bila statistički značajna.

DISKUSIJA

Uslovi u kojima se procenjuje rastvorljivost i poroznost materijala (*in vitro*) razlikuju se u pogledu veličine uzoraka, količine i vrste tečnosti u koju se uzorci potapaju, odnosno od dužine trajanja eksperimenta i vrste tečnosti koja se koristi [16]. U ovom istraživanju uzorci testiranih materijala potopljeni su u tečnost (destilovanu i dejonizujuću vodu) i čuvani su tokom 28 dana, što je u skladu sa metodologijom Gandolfija i sar., čime su praćene rastvorljivost i poroznost materijala u dužem vremenskom intervalu [17, 18].

Rezultati sorpcije tečnosti dobijeni u ovoj studiji nakon 28 dana u tečnosti pokazali su najmanju sorpciju tečnosti kod materijala MTA. Kod materijala KS izmerene vrednosti bile su nešto veće, dok je najveća sorpcija tečnosti uočena kod KAL. Ovakav rezultat bi se mogao objasniti veličinom čestica u sastavu materijala. Nanočestice u formulaciji KAL su manje, samim tim su izložene većoj površini, što može da dovede do izraženijeg kontakta između čestica materijala i površine na koju deluju. Veća reaktivna površina može da dovode do posledično većeg upijanja tečnosti, odnosno izraženijeg kontakta tečnosti sa ovim materijalom, što rezultira većom poroznošću materijala [19].

Dobijeni rezultati ukazuju na hidrofilnu osobinu svih testiranih cemenata, s obzirom na činjenicu da je zabeležena visoka sorpcija, odnosno poroznost materijala. Poroznost je osobina svih dentalnih cemenata koji se pripremaju mešanjem praha i tečnosti i posledica je inkorporiranja mehurića vazduha prilikom mešanja. Kod KAL cementa zabežene su veće vrednosti apsorpcije tečnosti, odnosno poroznosti u odnosu na MTA i KS, što ukazuje na činjenicu da materijal poseduje visoko poroznu strukturu.

Rastvorljivost i poroznost materijala su osobine materijala koje direktno mogu uticati na njihovu stabilnost, integritet i trajnost [20]. Poznato je da rastvorljivost i poroznost zavise od količine tečnosti koja se koristi prilikom pripreme cementa [21, 22], od vrste rendgenskog kontrasnog sredstva u kompoziciji materijala [23], kao i od vrednosti pH sredine [24].

Fridland i Rosado tvrde da svi dentalni cementi pripremljeni sa vodom imaju određen stepen poroznosti zbog inkorporacije mikroskopskih mehurića vazduha za vreme postupka mešanja cemenata. Isti autori tvrde da je prisutnost amorfnih pora i kapilarne strukture u kompoziciji MTA još jedan važan uzrok poroznosti ovog materijala [21].

Luz i saradnici tvrde da se poroznost KAL cementa smanjuje tokom vremena, što može da bude povezanao sa kontinuitetom formiranja i taloženjem hidratnih faza u novostvorenim porama [25, 26].

Ivone Regina de Oliveira i sardnici tvrde da je ključ fizičkih osobina rendgensko kontrastno sredstvo. Navedeni autori predlažu dodatak 15% ZnO : 10% Bi_2O_3 u kompoziciji KAL kao najpogodnije sredstvo koje može postići najbolji kompromis između dobrih fizičkih i mehaničkih svojstava i rendgenske kontrastnosti. Oni ukazuju da dodatkom 15% ZnO : 10% Bi_2O_3 dolazi do smanjenja poroznosti [27, 28, 29].

Isti autori tvrde da Bi_2O_3 povećava poroznost i smanjuje mehaničku čvrstoću KAL cementa čije su čestice različite veličine i izduženog oblika.

Garcia i saradnici su u svojoj studiji proveravali rastvorljivosti i poroznosti materijala na bazi KAL EB (EndoBinder) sa tri različita radiokontrastna sredstva: bizmut-oksid (Bi_2O_3), cink-oksid (ZnO) i cirkonijum-oksid (ZrO_2). EndoBinder je pokazao slično ponašanje kao MTA, bez obzira na rendgensku kontrastnost. Autori tvrde da dugo vreme vezivanja MTA-a dovodi do njegove nestabilnosti, odnosno do veće rastvorljivosti i dezintegracije, a posledično nastaje veće otpuštanje komponenata prisutnih u samom cementu. Rastvorljivost MTA iznosila je 5,74% za sivi, odnosno 6,65% za beli, i ove vrednosti su iznad graničnih vrednosti od (3%) predloženih specifikacijama [13].

Parreira i saradnici 2016. godine u svojoj studiji tvrde da dodatkom ZnO, kao i hidroksiapatita u formulacijama KAL cemenata dolazi do smanjenja nivoa poroznosti testiranih uzoraka i smanjenja pora posle kontakta sa simuliranim telesnim tečnostima. ZnO je biomaterijal sposoban da izazove mineralizacione procese [30].

Međutim, neki autori su mišljenja da velika rastvorljivost materijala može da bude i prednost, gledajući sa biološke i fizičko-hemijske tačke gledišta, jer dolazi do otpuštanja više kalcijumovih jona u okolno tkivo, isto kao što visok pH može da izazove veći antibakterijski efekat [30]. Samim tim, primenjeni u *in vivo* uslovima ovi cementi postaju izvor kalcijumovih i hidroksilnih jona sa posledičnom bioaktivnošću i antimikrobnim dejstvom.

Studije su pokazale da su joni kalcijuma glavna komponenta detektovana kod rastvorljivosti, pokazujući da je rastvorljivost cementa važan fenomen u otpuštanju kalcijumovih i hidroksilnih jona u periodontalno tkivo, što može da utiče na reparacijske procese [31].

Međutim, treba napomenuti da u kliničkim uslovima samo manji deo materijala dolazi u kontakt sa tkivnim fluidima, za razliku od laboratorijskih testova gde se čitav uzorak materijala potapa u tečnost, samim tim i osmotski efekat je izraženiji.

Štaviše, mora se uzeti u obzir da merenje razlika u težini uzoraka cementa takođe može zabeležiti procese raspadanja koji možda nisu rezultat rastvaranja. Na primer, čestice materijala mogu ispasti iz cementne strukture tokom skladištenja materijala u tečnosti tokom vremena [32].

ZAKLJUČAK

Rastvorljivost i apsorpcija tečnosti eksperimentalnog nanostrukturnog materijala na bazi KAL bile su značajno veće u poređenju su KS i MTA.