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Solubility and Porosity of New Nanostructured Calcium Silicate Cement

Tatjana Stanić[1], Violeta Pavlović[1], Vukoman Jokanović[2], Marija Živković-Sandić[3], Slavoljub Živković[1]

[1]Department of Restorative Dentistry and Endodontics, School of Dental Medicine, University of Belgrade, Belgrade, Serbia;
[2]Institute for Nuclear Sciences "Vinča", Belgrade, Serbia;
[3]Department of Orthodontics, School of Dental Medicine, University of Belgrade, Belgrade, Serbia

SUMMARY

Introduction Calcium silicate cements are most commonly used materials in endodontics for many indications due to their exceptional biological and physical properties. The aim of this study was to assess solubility and porosity of new experimental nanostructured calcium silicate cement.

Material and Methods A novel nanostructured calcium silicate cement (CS), commercialized calcium silicate cement (Biodentin), traditional glass ionomer cement (GIC) (Micron Superior) and resin-reinforced glass ionomer cement (Fuji VIII) were used in this study. All materials were prepared as per manufactuers' instructions, placed in metal rings of 16×2 mm diameter and kept in an incubator at 37°C for 24 hours. After that they were weighed and put in plastic containers filled with 25 ml of water for additional 24 hours. Then after all samples were weighed again and resorption and solubility were calculated.

Results The highest solubility was found for new CS cement (12.45) followed by conventional GIC Micron Superior (11.5) and Biodentin (6.1) whereas the lowest solubility was for resin reinforced GIC Fuji VIII (3.8). These differences were statistically significant ($p<0.005$). The highest absorption was also observed in new CS cement (24.15), followed by Biodentin (18.5) and Micron superior (17.95) while the lowest was for GIC Fuji VIII (7.75). These differences were also statistically significant except between Micron Superior and Biodentin.

Conclusion Solubility and porosity of new nanostructured calcium silicate cement were significantly higher than for traditional calcium silicate cement, traditional GIC and resin-reinforced GIC.

Keywords: solubility; porosity; new calcium silicate cement; glass ionomer cement; resin reinforced glass ionomer cement

INTRODUCTION

Calcium silicate based cements are most commonly used materials in endodontics for many indications due to their exceptional biological and physical properties [1, 2]. The best-known commercialized calcium silicate cement is Mineral Trioxide Aggregate (MTA), which is used for direct pulp capping, pulpotomy, apexification or closure of iatrogenic perforation and retrograde fillings in the apical surgery [3-7]. MTA powder consists of dicalcium and tricalcium silicate, tricalcium aluminate and tetracalcium aluminoferitte. Bismuth oxide is also added to this mixture in about 20% to achieve X-ray contrast [8, 9]. MTA is biocompatible material but it also has additional features such as bioactivity and bio-inductivity. Physical properties (adhesion, solubility, compressive strength) are satisfactory [1, 10, 11]. Due to chemical composition and long-lasting hydration, calcium silicate cements (and MTA) are slow-setting cements [12]. Long setting time of materials poses a significant risk of dissolution and leaching from the site of application whereas short working time (4 min) and granular consistency make clinical manipulation difficult [13].

Biodentin (Septodont, France) is tricalcium silicate cement with the addition of calcium carbonate and zirconium oxide that has become recently available on the market. Liquid component consists of calcium chloride (accelerator) and water, as a reducing agent. Changes in the chemical composition and applied technology have shortened the setting time of this material to 12 minutes [14]. In the recent years, an increasing number of new biomaterials intended to use in endodontic treatment and bone tissue engineering has been synthesized using nanotechnology [15]. Nanotechnology allows synthesis of materials that have particle size (1–100 nm) similar to the size of biomolecular structures with unique physical, chemical and biological properties [16]. High activity of nano-material particles accelerates their hydration and setting time [17].

Combining hydrothermal sol-gel method and method self-combusting waves experimental nanostructured calcium silicate cement (CS) was synthetized in the Institute

Address for correspondence: Violeta PAVLOVIĆ, Department of Restorative Dentistry and Endodontics, School of Dental Medicine, Rankeova 4, Belgrade, Serbia; *petrovic.violeta.bg@gmail.com*

of Nuclear Sciences in Vinca (Jokanović et al.). In addition to calcium silicate system (dicalcium and tricalcium silicate), which provides 60% of the total weight, fast setting gypsum and barium sulfate (X-ray contrast) are added (20% each). Setting of CS is 10 minutes after initial mixing with distilled water [18].

The aim of this study was to assess solubility and porosity of new experimental nanostructured calcium silicate cement.

MATERIAL AND METHODS

Experimental nanostructured calcium silicate cement (CS), commercialized calcium silicate cement (Biodentin, Septodont, France), conventional glass ionomer cement (Micron Superior, Prevest Dentpro Limited, India) and resin-reinforced glass ionomer cement (Fuji VIII, GC Corporation, Japan) were assessed in this study. Testing was conducted in accordance with the recommendations of ISO standards 6876. The new material was mixed with distilled water (3:1), while all other materials were prepared according to the manufacturer's instructions and then placed in metal rings of diameter 16×2 mm. For each material 6 samples were prepared. Tested materials were set during 24 hours in an incubator at 37°C, then after samples were removed from rings and measured at the scale (Acculab, group Sartorius, Goettingen, Germany) with precision of 0.0001 g. Obtained values are indicated as initial dry weight of the material (m_1). Samples were then placed (separately) in the plastic containers with 25 ml of distilled water. After 24 hours the samples were removed from distilled water, gently dried with filter paper weighed, and obtained values were marked as m_2. Samples were additionally dried using silica gel until constant weight was established (24 hours), which was marked as final dry weight of the material (m_3).

Absorption and solubility of materials were calculated according to the following formulas [19]:

$$\text{Absorption (\%)} = [(m_2 - m_3)/m_3] \times 100$$

$$\text{Solubility (\%)} = [(m_3 - m_1)/m_1] \times 100$$

RESULTS

The highest solubility was found for new CS cement (12.45) and Micron superior conventional GIC (11.05), then Biodentin (6.1) whereas the lowest value was observed in resin reinforced GIC Fuji VIII (3.8) (Table 1). The difference was highly statistically significant between Micron Superior and Fuji VIII and Biodentin as well as between Fuji VIII and Biodentin or Fuji VIII and new CS cement ($p<0.005$) and statistically significant between GIC Micron Superior and new CS cement ($p<0.05$).

The highest water absorption (porosity) was found for new CS cement (24.15), then Biodentin (18.5), Micron Superior (17.95) and the lowest for resin reinforced Fuji VIII (7.75) (Table 2). This difference was highly statistically significant between GIC Micron Superior and Fuji VIII and new CS cement, Fuji VIII and Biodentin, and Fuji VIII and CS cement or between Biodentin and CS cement ($p<0.005$). Satistically significant difference was observed between GIC Micron Superior and Biodentin ($p<0.05$).

Table 1. Solubility of tested materials
Tabela 1. Prosečne vrednosti rastvorljivosti ispitanih materijala

Material Materijal	N	Min	Mean	Max	SD
Micron Superior	6	10.5	11.05	11.7	0.46
Fuji VIII	6	3.5	3.8	4.2	0.30
Biodentin	6	5.6	6.1	6.6	0.37
CS Cement	6	11.7	12.45	13	0.51

N – number of samples; Min – minimum value; Mean – mean value; Max – maximum value; SD – standard deviation

N – broj uzoraka; Min – najmanja vrednost; Mean – srednja vrednost; Max – najveća vrednost; SD – standardna devijacija

Table 2. Liquid apsortion of tested materials
Tabela 2. Prosečne vrednosti poroznosti ispitanih materijala

Material Materijal	N	Min	Mean	Max	SD
Micron Superior	6	16.8	17.95	18.9	0.83
Fuji VIII	6	6.8	7.8	8.4	0.58
Biodentin	6	16.6	18.5	20.3	1.32
CS Cement	6	23.8	24.15	25.2	0.51

DISCUSSION

To estimate solubility and porosity of materials standard tests compiled in ISO 6876 were used. Weight difference of materials before and after storage in wet condition was key determinant for testing these characteristics [19-25]. As the main absorption of calcium silicate cements occurs in the first 24 hours [19, 26] and after that only minimal fluctuations in weight occurs, all materials were tested after 24-hours in wet condition.

Absorption of fluid in various degrees was observed in all tested materials indicating porous structure of tested cements. Porosity is characteristic of all dental cements prepared by mixing the powder with liquid as the consequence of air bubbles incorporation during mixing. In the case of calcium silicate cements, their specific amorphous structure significantly contributes to their porosity. Specifically, mixing powder with water forms porous calcium silicate hydrated gel that hardens over time. Pores within set cement are filled with water from the environment; therefore over time as cement sets, the number of pores is reduced [20].

In the current study, the most pronounced fluid absorption or porosity was observed in the experimental calcium silicate cement. Significant porosity of calcium silicate cements has been confirmed in numerous studies [19, 22, 27]. Also, the greatest solubility was observed in the CS cement. This is expected, as it is known that solubility of material is directly related to its porosity [14]. Main soluble fraction of calcium silicate cements is calcium hydroxide that in humid environment dissociates on calcium and hydroxyl ions. That way, *in vivo* applied cements become a source of calcium and hydroxyl ions with subsequent bioactivity and antimicrobial activity. However, balance between ion release and solubility is necessary in order to preserve cement integrity [28]. Substantially higher porosity of CS cement compared to Biodentin can be attributed primarily to their different chemical composition. In order to shorten the setting time, tricalcium silicate is the only phase used in the synthesis of Biodentin (dicalcium silicate not used). Also, the presence of calcium chloride and water as a reducing agent (polymer resin) in Biodentin could possibly affect the results. Obtained results can also be a consequence of different particle size of the tested materials. Smaller particles of nano calcium silicate cement (CS) and consequently their increased reactive surface could affect contact between liquid and particles, which could result in greater absorption of fluid in this material. It is also known that porosity and solubility of calcium silicate cements depend on the amount of liquid used for mixing the cement [20, 28], the type and the amount of X-ray contrast in their composition [26, 29] as well as pH (higher porosity is in the acidic environment [30]. Solubility and liquid absorption of glass ionomer cements also showed that chemical composition significantly affects physical properties. The presence of resin resulted in lower porosity and solubility of resin reinforced glass ionomer cements.

It is important to note that some limitations of standard tests could also affect results. For example, solubility of solid materials is determined by the amount of that substance that can be dissolved in a certain amount of solvent. Standard tests that are commonly used actually measure leaching of soluble material [1]. Also, in actual clinical settings, only a small part of material is in contact with moisture of the surrounding tissue whereas in laboratory conditions whole sample of material is in contact with large amount of liquid, where osmotic effect is more pronounced [20].

CONCLUSION

Solubility and porosity of new nanostructured calcium silicate cement were significantly higher than for traditional calcium silicate cement, traditional GIC and resin-reinforced GIC.

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Ispitivanje rastvorljivosti i poroznosti novosintetisanog nanostrukturnog kalcijumsilikatnog cementa

Tatjana Stanić[1], Violeta Pavlović[1], Vukoman Jokanović[2], Marija Živković-Sandić[3], Slavoljub Živković[1]

[1]Klinika za bolesti zuba, Stomatološki fakultet, Univerzitet u Beogradu, Beograd, Srbija;
[2]Institut za nuklearne nauke „Vinča", Beograd, Srbija;
[3]Klinika za ortopediju vilica, Stomatološki fakultet, Univerzitet u Beogradu, Beograd, Srbija

KRATAK SADRŽAJ

Uvod Cementi na bazi kalcijum-silikata danas su najčešće korišćeni materijali u brojnim endodontskim indikacijama zahvaljujući, pre svega, njihovim izuzetnim biološkim i odgovarajućim fizičkim svojstvima. Cilj ovog rada je bio da se proveri rastvorljivost i poroznost eksperimentalnog nanostrukturnog kalcijumsilikatnog cementa.

Materijal i metode rada Kao materijal u istraživanju korišćeni su novosintetisani nanostrukturni kalcijumsilikatni (CS) cement, komercijalni kalcijumsilikatni cement (Biodentin), konvencionalni glasjonomer-cement (GJC) Micron Superior i glasjonomer-cement ojačan smolom Fuji VIII. Svi materijali su nakon pripreme postavljeni u metalne prstenove promera 16×2 mm i čuvani u inkubatoru na 37°C tokom 24 sata, a zatim izmereni. Materijali su potom stavljeni u plastične kontejnere sa 25 ml vode i posle 24 časa posušeni, ponovo izmereni, a potom su utvrđivane apsorpcija i rastvorljivost.

Rezultati Dobijeni rezultati su ukazali na najveću rastvorljivost kod novosintetisanog CS cementa (12,45), potom kod konvencionalnog GJC Micron Superior (11,5), nešto manju kod Biodentina (6,1) a najmanju kod GJC ojačanog smolom Fuji VIII (3,8). Ove razlike su bile statistički značajne ($p<0,005$). Najveća apsorpcija je takođe uočena kod CS cementa (24,15), zatim kod Biodentina (18,5) i GJC Micron Superior (17,95), a najmanja kod GJC Fuji VIII (7,75). I ove razlike su bile statistički značajne, osim između GJC Micron Superior i Biodentina.

Zaključak Rastvorljivost i poroznost novosintetisanog nanostrukturnog kalcijumsilikatnog cementa je bila značajno veća u poređenju s komercijalnim cementom na bazi kalcijum-silikata, konvencionalnim GJC i GJC ojačanim smolom.

Ključne reči: rastvorljivost; poroznost; novi kalcijumsilikatni cement; glasjonomer-cement; glasjonomer-cement ojačan smolom

UVOD

Cementi na bazi kalcijum-silikata danas su najčešće korišćeni materijali u brojnim endodontskim indikacijama zahvaljujući, pre svega, njihovim izuzetnim biološkim i odgovarajućim fizičkim svojstvima [1, 2]. Najpoznatiji komercijalni kalcijumsilikatni cement je mineral trioksid-agregat (MTA), koji se koristi za direktno prekrivanje pulpe, pulpotomiju, apeksifikaciju, odnosno zatvaranje jatrogenih perforacija i retrogradnih kaviteta u apeksnoj hirurgiji [3-7]. Prah MTA se sastoji od trikalcijum i dikalcijum silikata, trikalcijum-aluminata i tetrakalcijum-aluminoferita. Radi postizanja rendgenske kontrastnosti, prahu je dodat bizmut-oksid u količini od približno 20% [8, 9]. Pored izražene biokompatibilnosti, MTA odlikuju i bioaktivnost i bioinduktivnost, kao i zadovoljavajuća fizička svojstva (adhezivnost, rastvorljivost, pritisna čvrstoća) [1, 10, 11]. Zahvaljujući hemijskom sastavu i dugotrajnoj hidrataciji, kalcijumsilikatni cementi (pa i MTA) su materijali koji se sporo vezuju [12]. Dugo vreme vezivanja predstavlja značajan rizik od rastvaranja i ispiranja materijala s mesta aplikacije i uz kratko radno vreme (četiri minute) i zrnastu konzistenciju čini ga relativno teškim za kliničku manipulaciju [13].

Odnedavno je na tržištu i Biodentin (*Septodont*, Francuska), trikalcijumsilikatni cement sa dodatkom kalcijum-karbonata i cirkonijum-oksida. Tečnu komponentu čine kalcijum-hlorid (akcelerator vezivanja) i voda, kao redukujući agens. Izmenama u hemijskom sastavu i primenjenom tehnologijom u sintezi ovog kalcijumsilikatnog cementa inicijalno vreme vezivanja je, prema specifikaciji, skraćeno na 12 minuta [14]. Poslednjih godina sve je više novih biomaterijala sintetisanih nanotehnologijom, namenjenih endodontskoj terapiji zuba i inženjerstvu koštanog tkiva [15]. Nanotehnologijom je moguće sintetisati materijale koji su, prema veličini čestica (1–100 nm), slični biomolekularnim strukturama, s jedinstvenim fizičkim, hemijskim i biološkim svojstvima [16]. Izražena aktivnost čestica nanomaterijala pospešuje njihovu hidrataciju i dovodi do bržeg vezivanja materijala [17].

U Institutu nuklearnih nauka u Vinči, prema recepturi V. Jokanovića i saradnika, kombinacijom hidrotermalne sol-gel metode i metode samosagorevajućih talasa, sintetisan je eksperimentalni nanostrukturni kalcijumsilikatni (CS) cement. Pored kalcijumsilikatnih sistema (dikalcijum i trikalcijum silikata), koji čine 60% ukupne mase, u pripremi materijala je korišćeno još 20% brzovezujućeg gipsa i 20% barijum-sulfata u svojstvu rendgenskog kontrastnog sredstva. Vezivanje materijala CS se potpuno završava nakon 10 minuta od početka mešanja sa destilovanom vodom [18].

Cilj ovog rada je bio da se proveri rastvorljivost i poroznost eksperimentalnog nanostrukturnog CS cementa.

MATERIJAL I METODE RADA

Testirani su eksperimentalni nanostrukturni kalcijumsilikatni (CS) cement, komercijalni kalcijumsilikatni cement Biodentin (*Septodont*, Francuska), konvencionalni glasjonomer-cement (GJC) Micron Superior (*Prevest Dentpro Limited*, Indija) i glasjonomer-cement ojačan smolom Fuji VIII (*GC Corporation*, Japan). Ispitivanje je obavljeno u skladu s preporukama ISO standarda 6876. Eksperimentalni materijal CS, zamešan sa destilovanom vodom (3:1), i kontrolni materijali, pripremljeni prema uputstvu proizvođača, postavljeni su u metalne prstenove promera 16×2 mm. Za svaki materijal napravljeno je po šest uzoraka. Nakon vezivanja materijala (24 časa) u inkubatoru na 37°C uzorci materijala su izvađeni iz kalupa i izmereni na vagici (*Acculab, Sartorius group, Getingen*, Nemačka) s preciznošću od

0,0001 g. Dobijene vrednosti su označene kao inicijalna suva masa materijala (m_1). Uzorci materijala su potom postavljeni (svaki posebno) u plastične kontejnere sa 25 ml destilovane vode. Posle 24 časa uzorci su izvađeni iz destilovane vode, blago posušeni filter-papirom i izmereni, a dobijene vrednosti označene su sa m_2. Zatim su isušeni silika-gelom do uspostavljanja konstantne mase (24 časa), koja je označena kao finalna suva masa materijala (m_3).

Apsorpcija tečnosti i rastvorljivost materijala izračunate su prema sledećim formulama [19]:

$$\text{Apsorpcija tečnosti (\%)} = [(m_2-m_3)/m_3] \times 100$$

$$\text{Rastvorljivost (\%)} = [(m_3-m_1)/m_1] \times 100$$

REZULTATI

Najveća rastvorljivost je uočena kod novosintetisanog CS cementa (12,45) i konvencionalnog GJC Micron Superior (11,05), potom kod Biodentina (6,1) a najmanja kod GJC ojačanog smolom Fuji VIII (3,8) (Tabela 1). Razlika u rezultatima je bila visoko statistički značajna između materijala Micron Superior i Fuji VIII i Biodentina, između Fuji VIII i Biodentina, odnosno Fuji VIII i novosintetisanog CS cementa ($p<0,005$), a statistički značajna između GJC Micron Superior i novosintetisanog CS cementa ($p<0,05$).

Najveća apsorpcija vode (poroznost) zabeležena je kod novosintetisanog CS cementa (24,15), potom kod Biodentina (18,5), nešto manja kod GJC Micron Superior (17,95), a najmanja kod smolom ojačanog Fuji VIII (7,75) (Tabela 2). Ova razlika je bila visoko statistički značajna između GJC Micron Superior i Fuji VIII, i novosintetisanog CS cementa, između Fuji VIII i Biodentina i Fuji VIII i novosintetisanog CS cementa, odnosno između Biodentina i novosintetisanog CS cementa ($p<0,005$). Statistički značajna razlika nije uočena jedino između GJC Micron Superior i Biodentina.

DISKUSIJA

Za procenu rastvorljivosti i poroznosti ispitivanih materijala primenjeni su standardni testovi objedinjeni u ISO 6876 kojima se pomenuta svojstva ispituju merenjem promena u težini materijala nakon njihovog čuvanja u tečnosti [19-25]. Kako se najveće upijanje tečnosti kod kalcijumsilikatnog cementa dešava u prva 24 časa [19, 26], a daljim boravkom u tečnosti dolazi do samo minimalnih oscilacija u težini, u ovom istraživanju svojstva materijala su ispitivana nakon njihovog 24-časovnog čuvanja u tečnosti.

Upijanje tečnosti u različitoj meri zabeleženo je kod svih ispitanih materijala, što ukazuje na inicijalno poroznu strukturu svih testiranih cementa. Poroznost je svojstvo svih dentalnih cementa koji se pripremaju s tečnošću i posledica je inkorporiranja mehurića vazduha tokom mešanja. U slučaju kalcijumsilikata, specifična amorfna struktura samih cementa značajno doprinosi njihovoj poroznosti. Naime, mešanjem praha s vodom formira se porozni kalcijumsilikatni hidratni gel koji vremenom očvršćava. Pore unutar vezanog cementa se ispunjavaju vodom iz okruženja, s tim da se vremenom i očvršćavanjem cementa broj pora smanjuje [20].

Najizraženija apsorpcija tečnosti, odnosno poroznost, u ovom istraživanju uočena je kod eksperimentalnog CS cementa. Generalno, znatno izražena poroznost kalcijum-silikata potvrđena je u brojnim studijama [19, 22, 27]. Takođe, najveća rastvorljivost je uočena kod materijala CS. Ovo je razumljivo jer je poznato da je rastvorljivost materijala direktno povezana s njegovom poroznošću [14], što je potvrđeno i u ovom istraživanju. Glavna solubilna frakcija kalcijumsilikatnih cementa je kalcijum-hidroksid, koji u vlažnoj sredini disosuje na kalcijumove i hidroksilne jone. Stoga primenjeni u uslovima *in vivo* ovi cementi postaju izvor kalcijumovih i hidroksilnih jona s posledičnom bioaktivnošću i antimikrobnim delovanjem. Ipak, balans između oslobađanja jona i rastvorljivosti je neophodan radi očuvanja integriteta cementa [28]. Značajno veća poroznost CS u odnosu na Biodentin može se pripisati najpre njihovom različitom hemijskom sastavu. Radi skraćivanja vremena vezivanja, u sintezi Biodentina nije korišćena dikalcijumsilikatna, već samo trikalcijumsilikatna faza. Takođe, prisustvo kalcijum-hlorida i vode kao redukujućeg agensa (polimerna smola) u Biodentinu mogli su uticati na dobijene rezultate. Ovi rezultati takođe mogu biti posledica različite veličine čestica ispitanih materijala. Manje čestice nanostrukturnog CS cementa i, posledično, njihova veća reaktivna površina mogle su uticati na izraženiji kontakt tečnosti sa česticama ovog materijala, što je moglo dovesti do veće apsorpcije tečnosti kod ovog materijala. Poznato je takođe da poroznost i rastvorljivost kalcijumsilikatnih materijala zavise i od količine tečnosti upotrebljene za pripremu cementa [20, 28], vrste i količine rendgenskog kontrasnog sredstva u njihovom sastavu [26, 29], kao i od pH vrednosti sredine, pri čemu su i poroznost i rastvorljivost izraženije u kiseloj sredini [30]. Iz rezultata rastvorljivosti i apsorpcije tečnosti GJC može se uočiti da i kod ovih materijala hemijski sastav značajno utiče na njihova fizička svojstva. Prisustvo smole dovelo je do manje poroznosti i rastvorljivosti GJC ojačanog smolom, što je, naravno, posledica njihovog hemijskog sastava.

Važno je napomenuti i da su određeni nedostaci standardnih testova mogli imati uticaja na dobijene rezultate. Naime, rastvorljivost čvrstih materijala se, prema definiciji, određuje količinom supstance koja se može rastvoriti u određenoj količini rastvarača. Testovi koji se uobičajeno koriste zapravo mere izlučivanje materijala rastvorljivih u vodi [1]. Takođe, u realnim kliničkim uslovima samo mali deo materijala je u kontaktu s vlagom okolnog tkiva, za razliku od laboratorijskih uslova, gde je ceo uzorak materijala u kontaktu s velikom količinom tečnosti, pa je ovde i osmotski efekat znatno izraženiji [20].

ZAKLJUČAK

Rastvorljivost i poroznost novosintetisanog nanostrukturnog kalcijumsilikatnog cementa su bile značajno veće u poređenju s komercijalnim cementom na bazi kalcijum-silikata, konvencionalnim GJC i GJC ojačanim smolom.