Compressive strength of calcium silicate-based cement

Vanja Opačić Galić1, Zoran Stamenić2, Violeta Petrović1, Vukoman Jokanović3, Slavoljub Živković1

1University of Belgrade, School of Dental Medicine, Department for Restorative Dentistry and Endodontics, Belgrade, Serbia;
2University of Belgrade, Faculty for Mechanical Engineering, Department for General Machine Design, Belgrade, Serbia;
3Vinca Institute of Nuclear Sciences, Department of Atomic Physics, Belgrade, Serbia

SUMMARY
Introduction The aim of this study was to compare compressive strength (Cs) of new nanostructural calcium silicate based cement (nCS) with commercial calcium silicate cement and conventional GIC.

Methods Four nanostructural materials were tested: nanostructural calcium silicate based cement (nCS) (Jokanović et al.), MTA Plus (Cerkamed, Poland), Fuji IX (GC Corporation, Japan) and Ketac Universal Aplicap (3M ESPE, USA). Five samples of each material were mixed in accordance with manufacturer's guidelines and positioned in metal moulds (ϕ4mm and 6mm). Compressive strength (Cs) expressed in MPa was determined after 24 hours, 7 days and 28 days respectively. Measurements were performed on universal testing equipment (Tinius Olsen, USA) at a crosshead speed of 1mm/min. For processing the results one-way ANOVA and post-hoc test were used.

Results The highest values of compressive strength after 24h was found in conventional GIC Fuji IX (mean 38.56±13.31) and Ketac Universal (mean 40.77±7.96). Calcium silicate cements after 24h showed low values of compressive strength (MTA Plus 5.91±0.28 MPa, nCS 1.35±0.36 MPa). After 7 days, FUJI IX 47.42±9.33 MPa and Ketac Universal 35.25±10.60 MPa showed higher value of compressive strength than MTA Plus (15.09±2.77 MPa) and nCS (11.06±0.88 MPa). After 28 days the Cs value for conventional GIC Fuji IX was 48.03±7.82 MPa and Ketac Universal 36.65±11.13 MPa while for calcium silicate cements it was 16.47±1.89 MPa and nCS 14.39±1.63 MPa. There was statistically significant difference (p<0.05) in Cs between conventional GIC and CS cements after 24h, 7 and 28 days.

Conclusions Calcium silicate cements initially showed lower values of compressive strength than conventional GIC that increased over time.

Keywords: calcium silicate cement; nanoparticle; glass ionomer cement; compressive strength

INTRODUCTION

Ideal material for root reparation should be able to close communication between the root canal and surrounding tissue, is biocompatible, dimensionally stable and insoluble when in contact with tissue fluids. The material is often placed in the root with an acidic environment, frequently with bacterial inflammation; therefore low pH level is an important factor that adds to the hardness and other properties of the cement [1]. In the past, materials such as calcium hydroxide, zinc oxide eugenol cements, resin composites, glass ionomer cements have been used for root canal perforation treatment but not all of them meet criteria of an ideal material [2].

GIC are developed by combining two different cements: silicate and zinc polycarboxylate cements [3]. Conventional GIC are made by an acid-base reaction of glass ions with a water solution of polyacrylic acid. They are considered potential biomaterials for orthopedic application because of their ability to adhere to bone and metals and good stability in wet environment. However, lack of bioactive potential and poor mechanical characteristics are some of the issues of this cement.

In the past few years, biocompatible calcium silicate hydraulic cements have been introduced in endodontic therapy. Mineral trioxide aggregate (MTA) is usually used as biomaterial for root and functional perforation reparation, as well as in other indications [4]. MTA is a bioactive material that forms an apatite layer on its surface when in contact with phosphates from tissue liquids but it also forms hybrid layer between dentin and calcium silicate materials [5]. It also releases some of its components in phosphate saliva puffers that encourage biomineralization processes [6]. There are number of calcium silicate cements on the market with the goal to surpass the deficiencies of the original formulation. MTA Plus is a nanostructural MTA released in 2012, with shorter binding time and lower concentration of heavy metals (up to 90%) in its formulation.

Nanoparticles allow uniform and homogenous structure, as well as lower temperature release while hydrating the cement (source: manufacturer). The use of nanoparticles has become an important research aspect in dentistry, with the focus on improving mechanical characteristics and antibacterial effect of the particles. The size of nanomaterial particles (<100nm) that is similar to the size of
biological molecules and structures (proteins, DNA, water) indicates possible uses in biomedical researches.

Newly synthesized nanostructural material used in our study uses tri-calcium and di-calcium silicates as a base. This calcium silicate system is produced with hydrothermal sol-gel method and self-expanding burning reaction [7], which secures its high activity and short bonding time. The smallest parts of this system are about 19.9 nm and show notable system activity [8, 9].

Compressive strength tests are used in dentistry for simulations of masticatory forces that clinically affect restoration or materials for covering or replacing tissue. The majority of masticatory forces are of compressive nature and their exact value is hard to determine.

The aim of this study was to test the compressive strength of a newly synthesized nanostructural CS cement and compare it to the commercial MTA Plus and conventional GIC that are used in functional or crown perforation reparations. The null hypothesis was that there was no difference in compressive strength between conventional and calcium silicate cements.

**MATERIAL AND METHODS**

Four cements were used in the research: nanostructural calcium silicate system (nCS) (Jokanovic et al.) where 60% of the total mass were β-C₂S i C₃S phases, 20% calcium carbonate and 20% BaSO₄ (Merck, Germany) as X-ray contrast. Water/powder mixing ratio was 1:2; MTA Plus (Cerkamed, Poland) hand mixed in the ratio 0,34 g distilled water and 1 g MTA powder; conventional GIC Fuji IX (GC Corporation, Japan), by the product instructions mechanically mixed in capsules for 10 seconds in an amalgamator; and self-adhesive and self-bonding GIC Ketac Universal Aplicap (3M ESP, USA), with glass oxides and liquid component that consisted of copolymer of acrylic and maleic acid and tartaric acid. As per manufacturer’s instruction the capsules were mechanically mixed for 10 seconds in an amalgamator at 4500 rpm.

**Sample preparation**

After mixing all materials were placed in two parted metal moulds 4 mm in diameter and 6 mm tall and they were condensed with a hand plunger, 5 samples for each material. The samples were kept on 37°C in a steam bath for 24h, 7 and 28 days. All cylindrical samples were polished with the finest abrasive paper and minimal pressure and visually checked. Samples with visible structural damage were eliminated from the study.

Compressive strength testing was done according to international standard ISO 9917-1:2007 (Dentistry-water-based cements- Part 1: powder/liquid acid-base cements) using a universal test machine (Tinius Olsen, USA; 5KN) at the speed of 1 mm/min along the longer axis of cylindrical samples [10]. The force needed to break the sample was noted and compressive strength in MPa was calculated with the formula Cs= 4P/πd where P was the maximum force needed to break the sample measured in N, and d was the diameter in mm.

For processing the results one-way ANOVA and post-hoc test was used. The level of significance was set at p<0.05.

**RESULTS**

The results are showed in the Tables 1-2 and Figure 1. The highest values of compressive strength after 24h were shown by conventional GIC Fuji IX (mean 38.56±13.31) and Ketac Universal (mean 40.77±7.96), but without sta-

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**Table 1.** Force (N) needed to break samples after 24 h, 7 and 28 days

|         | 24 hrs  |           | 7 days  |           | 28 days |           |
|---------|---------|-----------|---------|-----------|---------|-----------|
|         | MAX     | MIN       | MEAN    | SD        | MAX     | MIN       | MEAN    | SD        | MAX     | MIN       | MEAN    | SD        |
| nCS     | 24.05   | 12.15     | 16.91   | 4.54      | 149.10  | 121.50    | 138.94  | 11.08     | 197.15  | 145.20    | 188.90  | 18.38     |
| MTA     | 79.40   | 69.70     | 74.25   | 3.55      | 220.00  | 134.35    | 189.53  | 34.86     | 230.33  | 170.66    | 213.17  | 21.24     |
| Fuji IX | 675.17  | 289.33    | 484.37  | 167.13    | 688.00  | 423.67    | 595.00  | 116.70    | 543.30  | 321.83    | 512.67  | 87.90     |
| Ketac 3M| 593.17  | 399.00    | 512.07  | 99.96     | 569.17  | 234.00    | 442.77  | 133.09    | 569.00  | 224.00    | 381.17  | 125.00    |

**Table 2.** Compressive strength (Cs) values (MPa) of the tested materials in the function of time

|         | 24 hrs  |           | 7 days  |           | 28 days |           |
|---------|---------|-----------|---------|-----------|---------|-----------|
|         | MAX     | MIN       | MEAN    | SD        | MAX     | MIN       | MEAN    | SD        |
| nCS     | 1.35 ± 0.36 | 11.06 ± 0.88 | 14.39 ± 1.63 |
| MTA     | 5.91 ± 0.28 | 15.09 ± 2.77 | 16.47 ± 1.89 |
| Fuji IX | 38.56 ± 13.31 | 47.42 ± 9.33 | 48.03 ± 7.82 |
| Ketac 3M| 40.77 ± 7.96 | 35.25 ± 10.60 | 36.65 ± 11.13 |

**Figure 1.** Compressive strength values (MPa) in the function of time

nCS – nanostructural calcium silicate cement
nCS – nanostrukturni kalcijum-silikatni cement
tistical difference between them. Calcium silicate cements showed low values of compressive strength (MTA Plus 5.91±0.28 MPa and nCS 1.35±0.36 MPa), without statistical difference. A statistically significant difference was noticed between GIC and CS cements (p<0.05).

After 7 days, the highest compressive strength value was shown by FUJI IX 47.42±9.33 MPa and Ketak Universal 35.25±10.60 MPa, but without a statistically significant difference between them. The compressive strength of MTA Plus was 15.09±2.77 MPa and nCS 11.06±0.88 MPa, without statistically significant difference. There was a statistically significant difference between conventional GIC and CS cements (p<0.05).

After 28 days Cs value for conventional GIC Fuji IX was 48.03±7.82 MPa and Ketak Universal 36.65±11.13 MPa. There was no statistically significant difference between them. After four weeks, an increase in Cs value was noticed in calcium silicate cements, MTA Plus 16.47±1.89 MPa, and nCS 14.39±1.63 MPa but without statistically significant difference between them. Between the conventional GIC and CS cements there was a statistically significant difference (p<0.05).

**DISCUSSION**

Compressive strength is an indirect measure of bonding and strength of the material [11, 12]. It is an important property that may affect clinical performance [13]. This factor plays an important role in the treatment of functional perforations where cements are directly exposed to occlusal forces [14].

In the literature, significant variations in measured comprehensive strength have been reported as numerous factors can affect it. The cylindrical shape of the samples is convenient but sample surface perfection and intimate contact between samples and testing machine is hard to achieve [8]. Also, the size and shape of the samples, the preparation of the samples and hydration time, water/powder ratio, mixing technique, pressure while compacting, as well as the moisture and temperature of the room affect results [15, 16].

Conventional GIC are in wide use in clinical practice as cements or restorative materials. Many researches have been done with the goal to enhance mechanical and biological properties of GIC with incorporation of bioactive ceramic particles, glass powder and similar. Adding Zn has shown to have a stimulating effect on bone formation, as well as the moisture and temperature of the room affect results [11, 19]. As experimental conditions. That is how Akbari et al. [6] found that the Cs of White MTA (Angelus, Brazil) was 1.16 MPa after 24h and 2.19 MPa after 7 days, while Natale et al. [20] found Cs to be 18 MPa after 7 days. Noh et al. [21] found that WMTA (ProRoot MTA) after 24h had an average value of 19.41 and after 7 days 46.18 MPa, while Basturk et al. [16] showed results as high as 84.17 MPa after 4 days for ProRoot MTA. The microstructure and homogeneity of the cement affect its strength because finer particles have greater ability to absorb moisture.

Hand mixing of materials can result in inadequate hydration due to the limited formation of micropores inside the material that compromise water penetration in the material. Mitchell and Douglas pointed out that hand mixed cements have lower comprehensive strength due to trapped air, while capsulated cements mixed in a centrifuge have higher Cs [22, 16].

Nanostructural materials have particles that are not over 100 nm in size (most often between 5 and 50 nm), but therefore have up to ten times bigger surface area, which stimulates greater ettringate crystal formation [23]. Nanostructures strive to solve one of the key problems of endodontic cements like bonding time. Experiments indicate that in almost all nano-powders kinetic absorption and desorption can be improved simply by reducing the particle size [14].

Perfecting materials that can be used as biological bone “substitutes” is currently one of the most valuable and most active fields of biomaterial research. Biocompatibility and bioactivity of these materials secure the interaction with biological systems. Bioactive materials like calcium silicate cements, especially with nanostruc-
ture, stimulate regeneration of damaged tissue, therefore renewing the function of damaged tissue or organs [7].

CONCLUSION

The null hypothesis that there is no difference in Cs between conventional and calcium silicate cements is rejected. The compressive strength of conventional glass ionomer cements was significantly higher after 24 h, increased after 7 days and remained the same after 28 days. MTA Plus showed higher compressive strength after 24 h and 7 days than newly synthesized nanostructural calcium silicate cement (nCS) but the values were similar after 28 days. Compressive strength of calcium silicate cement grows with time and cement hydration.

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Ispitivanje kompresivne čvrstoće kalcijum-silikatnih cemenata

Vanja Opačić Galić1, Zoran Stamenić2, Violeta Petrović1, Vukoman Jokanović3, Slavoljub Živković1

Univerzitet u Beogradu, Stomatološki fakultet, Klinika za bolesti zuba, Beograd, Srbija;
Univerzitet u Beogradu, Mašinski fakultet, Katedra za opšte mašinske konstrukcije, Beograd, Srbija;
Univerzitet u Beogradu, Institut za nuklearne nauke „Vinča”, Laboratorija za atomsku fiziku, Beograd, Srbija

1Univerzitet u Beogradu, Stomatološki fakultet, Klinika za bolesti zuba, Beograd, Srbija;
2Univerzitet u Beogradu, Mašinski fakultet, Katedra za opšte mašinske konstrukcije, Beograd, Srbija;
3Univerzitet u Beogradu, Institut za nuklearne nauke „Vinča”, Laboratorija za atomsku fiziku, Beograd, Srbija

Ispitivanje kompresivne čvrstoće kalcijum-silikatnih cemenata

**MATERIJAL I METOD**

Testirana su četiri materijala – nanostrukturni CS (Jokanović i sar.), MTA Plus (Cerkamed, Poland), Fuji IX (GC Corporation, Japan) i Ketac Universal Aplicap (3M ESPE, USA). Po pet uzoraka za svaki materijal je zamešano po proizvođaču utiskivacima od 1 mm/min. Dobijeni rezultati su statistički obrađeni one-way ANOVA i post hoc Tukey’s testovima.

**ZAKLJUČAK**

Kalcijum-silikatni cementi su inicijalno pokazali niže vrednosti KČ u odnosu na konvencionalne GJC, ali su se one povećale u funkciji vremena. Ključne reči: kalcijum-silikatni cement; nanočestice; glasjonomer cement; kompresivna čvrstoća

**UVOD**

Idealan materijal za reparaciju oštećenja korena zuba treba da omogući zatvaranje komunikacije između kanala korena i okolnog tkiva, da je biokompatibilan, dimenziono stabilan i nerastvorljiv u kontaktu sa tkivnim fluidima. Materijal se često plasira u koren, gde je kiseloo kruženje, a često i sa bakterijskom inflamacijom, pa je nizak pH važan faktor koji utiče na tvrdoću i druga svojstva cemenata [1]. Dugi niz godina u terapiji perforacija kanala korena se koriste različiti materijali kao što su kalcijum-hidroksid, cink-oksid, gelatinomer cementi, kompozitne smole, glasjonomer cementi, ali nijedan u potpunosti ne ispunjava zahteve idealnog materijala [2].

GJC su razvijeni iz kombinacije dva različita cementa: silikatnih i cink-polikarboksilatnih [3]. Konvencionalni GJC nastaju acido-baznom reakcijom jona stakla sa vodenim rastvorom i poliakrilne kiseline i u širokoj upotrebi u kliničkoj praksi. Testovi kompresivne čvrstoće (KČ) koriste se u stomatologiji, sa fokusom na poboljšanje mehaničkih osobina i napredno kruženje i nadoknadu tkiva. Većina mastikatornih sila su kompresivne prirode i njihovu tačnu vrednost je teško utvrditi [8, 9].

Poslednjih godina se u endodontski terapiju uvode nanomaterijali: nanostrukturni kalcijum-silikatni sistem (nCS) (Jokanović i sar.) sadrži 60% ukupne količine β-C2S i C3S faza, 20% kalcijske i 20% BaSO₄ (Merck, Nemačka) kao rendgen}

**MATERIJAL I METOD**

U istraživanje su uključena četiri cementa: Nanostrukturni kalcijum-silikatni sistem (nCS) (Jokanović i sar.) sadrži 60% ukupne količine β-C₂S i C₃S faza, 20% kalcijum-karbonata i 20% BaSO₄ (Merck, Nemačka) kao rendgen
Contrast is not good, so I will try to make the content more readable:

Contrast s Weekend. Phra is measured on a distillate medium water in amounts 1:2 water:phra.
MTA Plus (Cerkamed, Polska) is used in amounts 0.34 g distillate water in 1 g phra MTA.

Conventional GJC Fuji IX (GC Corporation, Japan) - the pre-processing test produces a mechanical response that is measured in capsules 10 seconds in the amalgamator at 4500 rpm.

The adhesive and adhesive GJC Ketak Universal Universal (3M ESP USA), so that the pigments are free of agglomerates, and the color component is a polycrystal abrasive paper and visking silex. The pre-processing of the test capsules are mechanical measurements 10 seconds in the amalgamator at 4500 rpm.

Preparation of specimens

Materials were used for measurements of fluctuating in a number of metal cores 4 mm and 6 mm, where the low denominated rubber nailing, at the time the material. Lower side are drawn on 37 C in a paraffin cup at 24, 7 and 28 days. Sili cilindrically lower side are polished with the most fine abrasive paper and visually proven, and lower side were cleaned in the structures are sanded.

Testing the KČ from the test materials is performed in the following, under commonly accepted standard ISO 9917-1:2007 (Dentistry-water-based cements - Part 1: powder/liquid acid-base cements) [10]. KČ is measured on a universal test machine (Tinius Olsen, USA 5KN) with a brinell unit for 1 mm / min. below the sili cilindrically lower side. Silica is available to polycrystal lower side is selected and KČ measurement is made in MPa, where G is the maximum force is measured as a plate lower side is selected, and KČ is measured in MPa is calculated from the formula $K_C = 4P / \pi d^2$, where $d$ is the diameter of the test specimen.

Research KČ test materials is being performed in a solution of the formula.

With the test pressure of 1 mm / min. along the axis and 2KN (USA 5KN) lower side is selected. The test results are shown in Table 1 and 2 and in Figure 1.

In all the results are presented for one-way ANOVA and post-hoc test. Significance level is determined as $p < 0.05$.

**RESULTS**

Obtained results are presented in Tables 1 and 2 and in Figure 1.

Maximum values of KČ are presented in Table 1 and 2 for 24 hours, and 2.19 MPa after seven days, while Natale et al. [6] find that KČ for White MTA (Angelus, Brazil) is 1.16 MPa during the first day, and the rest is done for the remaining 12 months. This is an indicator of the quality of the material and is important in the formulation of the active agent.

Observations are made on the basis of the obtained results and the properties of the material, and in the nanoformulation, it is shown to have a significant activity [3]. Titanium dioxide is added to increase cell proliferation [3]. The test results show that MTA Plus (Cerkamed, Poland) has the highest KČ value, followed by Ketak Universal (3M ESP USA), and the lowest value is shown by the test material.

**DISCUSSION**

KČ is indirect measure of the formation and subsequent material [11, 12] and is important in the formulation of the active agent. The agent is used in the clinical performance of the material [13]. The agent produces a significant ulterior effect on the formation of the material, and is used in the clinical performance of the material [14].

In literature there are many variables in the results, where on the basis of the obtained results, the agent produces a significant ulterior effect on the formation of the material, and is used in the clinical performance of the material [14].

Conventional GJC are used in the upper area of the clinical practice as cement or as restoration materials. Many studies show that the production of calcium hydroxide and biological structures is significant. The production of calcium hydroxide and biological structures is significant. The test results are shown in Table 1 and 2 and in Figure 1.
[21] nalaze da je WMTA (ProRoot MTA, USA) posle 24 sata imao prosečne vrednosti 19,41, a posle sedam dana 46,18 MPa, dok Basturk i sar. [16] iznose vrednosti i do 84,17 MPa posle četiri dana za ProRoot MTA. Mikrostruktura i homogenost cementa utiču na njegovu čvrstoću, jer sitnije čestice imaju veću sposobnost apsorpcije vlage.

Ručno mešanje i unošenje materijala takođe može uticati na neadekvatnu hidrataciju zbog ograničenog formiranja mikropora unutar materijala, što kompromituje prodor vode u hidrat materijala. Mitchell i Douglas ističu da ručno mešani cementi imaju slabiju čvrstoću, zbog zarobljenog vazduha, dok inkapsulirani cementi koji se mešaju i centrifugiraju imaju veću KČ [22, 16].

Nanostrukturni materijali imaju čestice koje ne prevazilaze veličinu od 100 nm (najčešće između 5 i 50 nm), ali zato imaju i do deset puta veću interaktivnu površinu, što utiče na povećano formiranje etringit kristala [23]. Nanostrukture pokušavaju da reši jedan od ključnih problema endodontskih cemenata kao što je vreme vezivanja. Eksperimenti ukazuju na to da kod praktično svih nanoprahova kinetika apsorpcije i desorpcije može biti unapredena jednostavno smanjenjem veličine zrna [14].

Usavršavanje materijala koji bi mogli da se koriste kao biološke „zamene“ kosti danas je jedna od najznačajnijih i najakтивnijih oblasti istraživanja biomaterijala. Biokompatibilnost i bioaktivnost ovih materijala obezbeđuje interakciju sa biološkim sistemima. Bioaktivni materijali kakvi su kalcijum-silikatni cementi, posebno sa nanostrukturom, stimulisu regeneraciju oštećenih tkiva, a time i obnavljanje funkcije oštećenih tkiva ili organa [7].

ZAKLJUČAK

Nulta hipoteza da nema razlike u KČ između konvencionalnih i kalcijum-silikatnih cemenata je odbačena. KČ za konvencionalne glasjonomer cemente je bila značajno viša posle 24 sata, a rasla je posle sedam dana i ostala ista 28 dana. MTA Plus je pokazao veću KČ posle 24 sata i sedam dana u odnosu na novosintetisani nanostrukturni kalcijum-silikatni cement (nCS), ali ove vrednosti su se izjednačile posle 28 dana. KČ kalcijum-silikatnih cemenata značajno raste u funkciji vremena i sa hidratacijom cemenata.