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"The Evaluation Nano Calcium Silicate Cements Performance for Palpation"

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Abstract:

Root canal therapy includes cases of dental treatment that causes complications such as infectious of bone. Therefore, a substance that can also combined excellent sealing with appropriate tissue response is necessary.

In recent years, studding on the addition of various calcium silicate cement base for making these cements bioactive as a root canal filling materials is done ; however, influence of addition tri-calcium phosphate on the reform Bioactivity and other properties of these cements in this article was not research yet, which revealed that adding calcium phosphate significantly affects the properties of silicate cement in a positive way.

After the cement powder components were mixed with double distilled water, complex physical and chemical reactions happen that in this reactions, the silicates of the cement products hydrations and with solving phosphate compounds and precipitate calcium phosphate such as hydroxyapatite, a hard solid mass produce.

SEM images of composite cement shows the spherical particles of hydroxyapatite after one day immersed in SBF. The results showed that calcium phosphate phase is a resource for encourage and accelerate the apatite layer on the surface of the composite cement. The researchers reported that the hydrolysis of α-TCP after 15 days was completed.

As a result, cement made in this study with more tests can used for dental procedures. This cement has ability to setting itself, high strength, biocompatibility and the ability of inject.

Key words: nano calcium silicate, cement, composite, biocompatibility

INTRODUCTION:

Regeneration of bone defects caused by trauma, infection, tumors or inherent genetic disorders is a clinical challenge. Autologous bone or autograft is still considered the clinical “gold standard” and the most effective method for bone regeneration as it promotes bone formation over its surface by direct bone bonding (osteoconduction) and induces local stem cells to differentiate into bone cells (osteoinduction) without any associated immune response.[1,2]

In autologous bone grafting fresh cortical or trabecular bone or a combination of both, are transplanted from one site in the body, such as the iliac crest, to another within the same patient. However, autografting presents several disadvantages; with limited bone supply and donor site morbidity being the major drawbacks. These disadvantages can be overcome by using allograft.[1]

An alternative to autografts is the use of allografts (tissue from another patient), which accounts for 34% of the current bone grafts. This also has limitations including availability, disease transmission and risks of infection. The limitations of these two approaches can be overcome by the use of synthetic bone substitutes/scaffolds.[3]

Table 1 summarizes the different groups of bone substitutes: natural and synthetic biodegradable polymers, ceramics including bioglasses, metals and composites. All of these materials have advantages as well as disadvantages which could be overcome by combining different materials. (Table 1).[1]

Native bone tissue is a composite composed of hydroxyapatite (HA) crystals (2–5 nm wide and 70 nm long) and collagen fiber matrix (50–500 nm diameter).Hence polymer–ceramic composites are considered as ideal material for bone scaffolds. However, nanocomposites are believed to be more advantageous as they have better mechanical properties and high cell-surface interaction.[3]

MATERIALS AND METHODS:

1-1 Sample preparation

Initially, the combination of α-tricalcium phosphate and β-tricalcium phosphate were added to tricalcium

TABLE 1. BONE GRAFTING MATERIALS USED FOR BONE REPAIR AND REGANARATION: EXAMPLE, ADVANTAGES AND DISADVANTAGES[1]

Bone grafting materials		Examples	Advantages	Disadvantages
Polymers	Natural	– Protein: collagen, fibrin, gelatin, silk fibroin – Polysaccharides: hyaluronic acid, chondroitin sulphate, cellulose, starch, alginate, agarose, chitosan, pullulan, dextran	– Biodegradability – Biocompatibility – Bioactivity – Unlimited source (some of them)	– Low mechanical strength – High rates of degradation – High batch to batch variations
	Synthetic	– Poly-glycolic acid (PGA) – Poly-lactic acid (PLA) – Poly-(ε-caprolactone) (PCL) – Poly-(lactide-*co*-glycolide) (PLGA) – Poly-hydroxyethylmethacrylate (poly-HEMA)	– Biodegradability – Biocompatibility – Versatility	– Low mechanical strength – High local concentration of acidic degradation products
Ceramics	Calcium-phosphate	– Coralline or synthetic hydroxyapatite (HA) – Silicate-substituted HA – β-Tricalcium phosphate (β-TCP) – Dicalcium phosphate dehydrate (DCPD)	– Biocompatibility – Biodegradability – Bioactivity Osteoconductivity	– Brittleness – Low fracture strength – Degradation rates difficult to predict
	Bioglasses and glass-ceramics	– Silicate bioactive glasses (45S5, 13-93) – Borate/borosilicate bioactive glasses (13-93B2, 13-93B3, Pyrex®)	– Osteoinductivity (subject to structural and chemical properties)	
	Others	– Alumina ceramic (Al_2O_3)		
Metals		– Titanium and its alloys – Tantalum – Stainless Steel – Magnesium and its alloys	– Excellent mechanical properties (high strength and wear resistance, ductility) – Biocompatibility	– Lack of tissue adherence – Corrosion – Risk of toxicity due to release of metal ions
Composites		– Calcium-phosphate coatings on metals – HA/poly-(D,L-lactide) – HA/chitosan-gelatin	– Combination of the above	– Combination of the above

silicate cement in different weight percentages and based on the measurement of setting time and compression strength test, the optimum values of this combination which were 10% of α-tricalcium phosphate and 10% of β-tricalcium phosphate, were chosen. Then Nano-HA compound was added to the optimum sample by 1% of weight.

All of the tests in this part were carried out by one person. During the mixing, the USA SSW spatula was used. All the used equipment to carry out the work, were autoclaved and mixed with the ratio of 3 to 1 by bedistilled water.

1-2 Tri-Calcium Silicate powder

Tri-Calcium silicate powder was synthesized from $(C_2H_5O)_4Si$, $Ca(No_3)_2$ $4H_2O$ precursors and nitric acid by sol-gel method. Sequences and various amounts of raw material were examined and analyzed and the optimum gel was determined which the synthesis process was the way that initially by adding certain amount of bedistilled water to $Si_4(C_2H_5O)$ and then adding nitric acid, solution was mixed by an electrical mixer for 1 hour. Entirely, $Ca(NO3)_2$ $4H_2O$ was added to the mixture and after 1 hour mixing, the optimum sol was achieved. The optimum sol was placed at 60°c for 24 hour and then at 120°c was dried and after being placed at 1450°c for 6 hours, it was quench-calcinated. The produced calcium silicate powder has very high purity (99%) and contained nano-particles and micro-particles which was examined by FTIR and XRD analysis.

1-3 HA Nano particles

HA Nano particles were synthesized by sol-gel method. First, TEP in ethanol was hydrolyzed in the presence of small amount of distilled water and was placed on the mixer for 24 hours. Appropriate amount of TEP solution was added to calcium nitrate solution and it was placed on the mixer for 1 hour. Then it was placed at 25°c for 1 day and again it was place at 40°c for 3 days. Finally, achieved powder was examined by conducting TEM analysis.

1-4 α and β tri-calcium phosphate

First, tri-calcium phosphate powder was purchased from Merck company and then to obtain α-tricalcium phosphate powder, the purchased powder was placed at 1350°c for a certain time and to obtain β-tricalcium phosphate powder, the purchased powder was placed at 1250°c for a certain period of time. After heat treatment, the resulting powders were immediately cooled in the air. Then, XRD analysis was carried out on the resulting powders to

assure that the obtained powder possesses α-tricalcium phosphate and β-tricalcium phosphate.

1-5 FTIR test

Structural variations of all sample powders were examined at constant weight value by using FTIR and within the spectrum range of 500 - 4000 cm^{-1}.

1-6 SEM test

In order to evaluate morphology and surface structure, a scanning electron microscope (SEM) was used. In this study, samples were evaluated by SEM after gold coating. In order to ensure consistency and uniform distribution of particles, an electron microscope by using elements distribution detector (Map) and existing Quantex software at RAZI research center, were used.

1-7 Hardening time measurement test

The first intended test in this thesis, is the test to determine the cement hardening time based on ASTM C266 standard. For this purpose Gilmore device was used. Basis of this device is that a force with certain weight and determined diameter is applied to cement in a pre-determined dimensions moll and the impact of a needle on it is checked.

According to ISO6876 standard, timing during the preparation of cement paste is divided into three parts:

1- Mixing time: the mixing time which is calculated since the liquid contact with cement powder that this time for silicate-based dental cement is about 50 seconds.

2- Working time: time that cement should be transmitted to its intended location.

3- Hardening Time: is called to the time that cement becomes hard or during applying load by Gilmore device, no trace of Gilmore needle stands on cement.

1-7-1 Required instruments for measuring hardening time

1- Incubator with 37±1 °c and relative humidity of over 95%

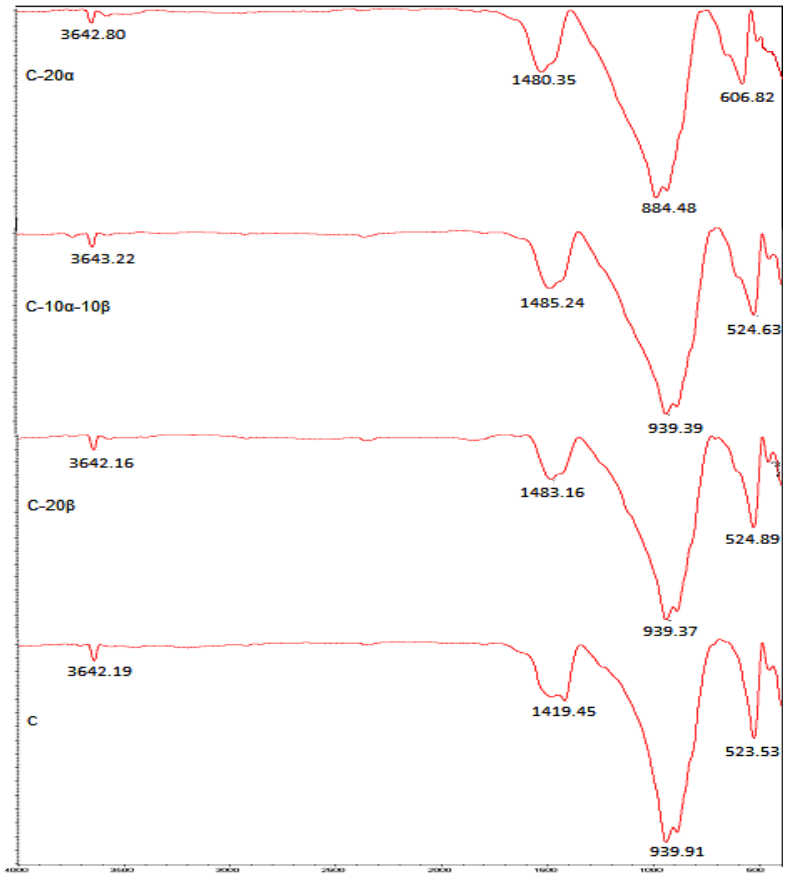

Figure 1. FTIR from tri-calcium silicate based cements.

2- A needle penetrating in cement with 2.12 mm and 1.06 mm and in cylindrical shape.
3- Applied force with the weight of 113.4 gr and 453.6 gr.

DISCUSSION AND CONCLUSION

2-1 Structural variations

Structural variations of sample cements, were examined by using FTIR and these variations can be observed in figure 1.

2-2Results for hardening test

Primary and secondary results of hardening test are shown in table 2. By adding HA nano compound to C-10α-10β, the initial hardening time increased. By adding α-tricalcium phosphate to C cement it was observed that hardening time decreased and also by adding β-tricalcium phosphate, hardening time increased.

By adding HA Nano compound to C-10α-10β dental cement, the primary hardening time slightly increases. According to hardening time and extreme stability of HA, it seems that HA particles do not participate in hydration process and causes delay in hardening time. Phosphate calcium compounds such as α-tricalcium phosphate have short primary hardening time between 10-15 minutes by themselves. Therefore, by adding phosphate calcium compound to silicate-calcium cement, hardening time still remains short which of course this is the opposite for β-tricalcium silicate.

2-3 Results and SEM test analysis

Morphology of C hydrated cement samples after immersion in SBF are shown in below figures. Hydroxiapatite crystal sediments can be clearly seen on cement surface. Figure 2 shows SEM image from C-10α-10β-1HA after being placed in SBF for 1 day at different magnifications. Adding calcium phosphate compound to tri-calcium silicate causes rise of bio-activity. This is because of phosphate calcium compounds cause a rise in bio-activity by themselves. Bio-activity evaluation (ability to form chemical bond with the living bone tissue) takes place through the formation of apatite layer on cement surface.

Apatite formed on cement surface in most cases is non-stoichiometric (Ca_{10-x} $(PO_4)_3(OH)_{2-x}$) which has calcium deficiency, similar to what is found in natural bone when C-10α-10β cement is placed in SBF, calcium hydroxide is formed in portlandite shape. OH^- ion release causes PH increase. Increasing PH value and calcium ion release, leads to the formation of apatite layer on cement surface. Also, functional groups such as Si-OH act as place of modifiers for hydroxyl apatite sediment. By comparing C-10α-10β-1HA cement to C-20β cement it can be found that the existence of TCP compound is more effective than CDH compound to create hydroxyl apatite phase on cement surface which represents the increase in bio-activity. In bone calcification, first amorphous calcium phosphate phase (ACP) is formed as intermediate phase on cement surface, according to PH≥9, ACP

TABLE 2. HARDENING TIME FOR DENTAL CEMENT SAMPLES

Secondary hardening time (minutes)	**Primary hardening time (minutes)**	**Examined sample**
60	13	C
30	14	C-20α
35	14	C-15α-5β
35	8	C-10α-10β
35	10	C-10α-10β-1HA
70	30	C-20β

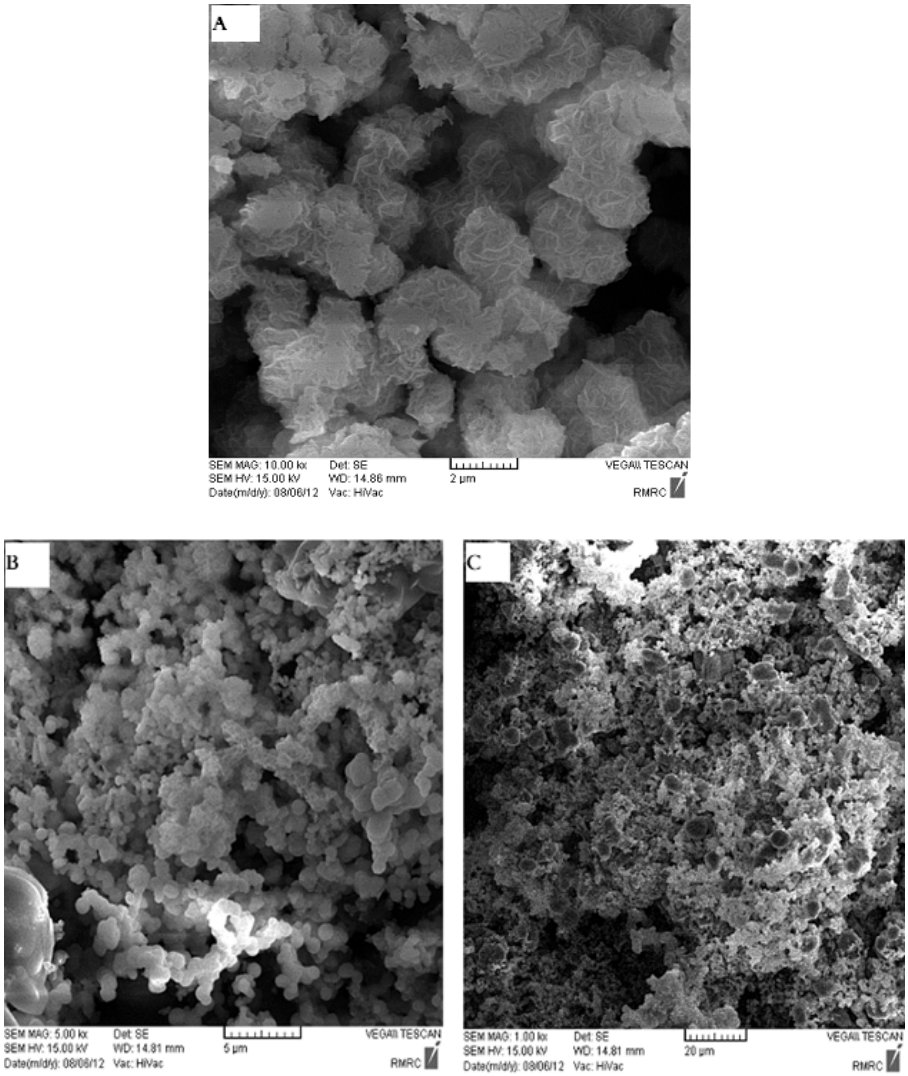

Figure 2. Surface morphology from C-10α-10β-1HA hydrated cement after a day immersion in SBF

A: Magnification*10000, B: Magnification*5000, C: Magnification*1000

phase turns to hydroxyapatite phase by passing through octacalcium phosphate phase. In addition to creation of apatite layer on cement layer, calcite is also formed. Creation of apatite and calcite layer, prevents ionic penetration and as a result it leads to reduction of destruction phenomenon and rising the sealing property of the made cement. In examining conducted FTIR's it was determined that the apatite structure is observed in all three C-20α, C-10α-10β and C-20β samples with (1480-1490 cm^{-1}) P-O vibrating models. A small pick is also seen within the range of 3640-3645 cm^{-1} which is related to OH-stretch mode. As it was observed, this pick has not changed by variation of existing calcium phosphate compound in cement. Also, 606 cm^{-1} is related to OH vibration mode for apatite hydroxyl which is obviously clear in C-20α that has gradually decreased by reduction of α-tri calcium phosphate amount the way so that it has almost faded in C-20β sample. Intense and broad 939cm^{-1} pick which is clearly visible in C-10α-10β, 20β and C samples is related to Si-O that by reduction of α-tricalcium phosphate percentage existing in composite cement its width has reduced and in cement C, the greatest depth has been reached for the mentioned pick. On the other hand, in C-20α sample a great pick is observed at 884cm^{-1} which is related to PO_4^{3-} and meanwhile it seems that the pick related to Si-O in this sample, has slightly shifted towards higher frequencies and finally it has increased intensity and width of PO_4^{3-} pick.

As a result, the made cement in this project can be applied for dental affairs by more tests and their verifications. This cement has the hardening ability by itself and it has high strength, bio-compatibility and injectivity.

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