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Primary research to put bioactive glass into toothpaste

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Abstract
This paper presents the synthesis of bioactive glass $40SiO_2$-40CaO-$20P_2O_5$ (wt. %) using the sol-gel technique. XRD analysis demonstrates that the material can successfully synthesized by the dried gel at 750 ºC for 3 hours. *In vitro* experiment confirmed the bioactivity of synthetic glass by the formation of hydroxyapatite (HA) layer on its surface after 15 days immersed in SBF solution. Thanks to the bioactivity, synthetic glass was added to the toothpaste to create a smart product which is capable of restoring surface defects. SEM observation shows that the surface of tooth sample brushed with toothpaste containing bioactive glass was filled without defects after 1 month of testing.

Keywords: bioactive glass, bioactivity, hydroxyapatite, *in vitro*, toothpaste

Introduction
Bioactive glasses are used as artificial materials to repair and fill defective bones [1]. These glasses are composed of oxides such as CaO, SiO_2, P_2O_5, Na_2O, which are linked together to form amorphous lattice of materials. The bioactivity of bioactive glasses is the ability to form a mineral layer of hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$ (HA) on the surface when they are immersed in a physiological solution or implanted directly in the human body. The hydroxyapatite (HA) mineral layer is identical to the inorganic component of human bone, so it is the connecting bridge between the graft made from glass material and natural bone [1, 2].

In the past 50 years, different bioactive glasses have been synthesized, and studied for applications in bone implants, making dental fillings. A number of products are currently commercialized, and applied in bone grafting techniques, dental fillings, or as Ca supplements [3, 6].

Thanks to the bioactivity property, bioactive glasses are initially added to toothpaste for the purpose of restoring blemishes, scratches on the surface of teeth [7]. We can list several types of toothpastes containing bioactive glass's materials on the market today such as Sensodye, BioMin. However, there are very few scientific reports on putting bioactive glass to toothpaste to repair defective surface teeth.

This work presents the synthesis and characterization of bioactive glass $40SiO_2$-40CaO-$20P_2O_5$ as well as the addition of this material to hand-make toothpaste to create a smart product that can restore the defects, and scratches on the teeth surface.

Materials and Methods
Materials
Chemicals with over 99% purity purchased from Sigma-Aldrich are listed as calcium nitrate tetra-hydrate $Ca(NO_3)_2.4H_2O$; tetraethyl ortho-silicate $Si(OC_2H_5)_4$ (TEOS); tri-ethyl phosphate $(C_2H_5)_3PO_4$ (TEP); nitric acid HNO_3; sodium bicarbonate $NaHCO_3$; sodium lauryl sulfate surfactant $CH_3(CH_2)10CH_2(OCH_2CH_2)_nOSO_3Na$; Glycerin $C_3H_5(OH)_3$; coconut oil; menthol.

2.2. Synthesis of bioactive glass $40SiO_2$-40CaO-$20P_2O_5$ (wt. %)
The bioactive glass system $40SiO_2$-40CaO-$20P_2O_5$ was synthesized by sol-gel method. The synthesis process is presented as follows. Firstly, the amount of 20 mL of distilled water was placed into a glass reaction vessel. The Reaction system was adjusted to an acidic medium at pH = 2 using HNO_3 acid which acts as a catalyst for the hydrolysis of TEOS and TEP alkoxides. After that, TEOS, TEP and Ca $(NO_3)_2.4H_2O$ were consecutively put into the reaction vessel. A magnetic stirrer was used to stir the reaction mixture during the whole synthetic process. After the substances dissolved completely, we obtained a homogeneous bright white sol solution. Next, sol solution was left for 5 days at a temperature of 70 ° C to condense into gel. The obtained gel was dried at 100 ° C for 24 hours to completely remove the solvent, and obtain a powder product. Finally, the powder product is heated at a temperature of 750 ºC for 3 hours to decompose Ca $(NO_3)_2.4H_2O$ salt into CaO. This step gives the Ca^{2+} cations from CaO oxides which act as breaking agents of the orderly structural bonds -Si-O-Si-, forming an amorphous structural lattice of glass.

Synthesis of toothpaste
Toothpaste was made by mixing a mixture of 30 g of sodium bicarbonate $NaHCO_3$; 15 g of coconut oil; 5 g sodium lauryl sulfate; 5 g of glycerol; 10 g H_2O; 2 g of bentonite; and 2 g of peppermint oil for 30 minutes. The resulting toothpaste product is pure white in color, in a viscous form.

In the above ingredients, $NaHCO_3$ is responsible for creating porous, bleaching teeth; coconut oil for polishing teeth and preventing bacteria; sodium lauryl sulfate used as a surfactant, enhancing teeth whitening ability; glycerin for lubricating, moisturizing and sweetening effect; bentonite for providing natural mineral particles, helping to remove food plaque better when brushing teeth; peppermint essential oil used for both antibacterial and a refreshing fragrance of toothpaste.

For toothpaste containing bioactive glass, an amount of 5% by weight of sol-gel bioactive glass was added to the mixture and mixed with the same process as above. Samples of toothpaste were stored in sealed glass jars and stored at room temperature.

In vitro experiment for bioactivity assessment

The *in vitro* test was carried out to check the bioactivity of synthetic glass through the possibility of forming new bone mineral. The experiment was conducted by soaking glass powder in the simulated body fluid (SBF) solution with ionic composition similar to blood in the human body [8]. Glass powder sample was soaked in SBF solution and kept at 37 °C with the stirring speed of 50 (rpm). After immersion interval, the glass powder was separated, washed with distilled water to remove excess ions, and then rinsed with pure alcohol to completely remove the free ions. Powder sample was dried, and stored for chemical-physical investigation.

Activity of toothpaste containing bioactive glass

Human teeth sample was tested with toothpaste containing bioactive glass to evaluate the ability to recover surface defects. Teeth sample was brushed with the toothpaste containing bioactive glass three times a day at morning, noon, and evening. The experiment was continuously conducted for 1 month. After finishing, the tooth sample was measured for surface morphology using SEM method, and compared with the tooth surface at the beginning.

Results and discussions

Characterization of bioactive glass before and after *in vitro* experiment

Figure 1 shows XRD diagrams of glass samples before and after *in vitro* experiments. The synthetic glass sample did not have any characteristic peak for the crystalline phases, which is characteristic for material with an amorphous structure [9, 10]. After *in vitro* experiment for 15 days, XRD diagram of the glass system appeared characteristic lines for crystal phases. Specifically, two evident diffraction peaks at 32 and 45° (2θ) were assigned to hydroxyapatite (HA) phase, corresponding to miller planes (211) and (222) (JCPDF. 90432). This result confirmed the bioactivity of synthetic glass material after *in vitro* experiment.

In addition, peaks characterizing $Ca_3(PO_4)_2$ phase with low intensity were observed. According to the references [11, 12], $Ca_3(PO_4)_2$ compound is also a bioactive material. In the physiological environment, $Ca_3(PO_4)_2$ material is dissolved and re-precipitated gradually, creating HA phase.

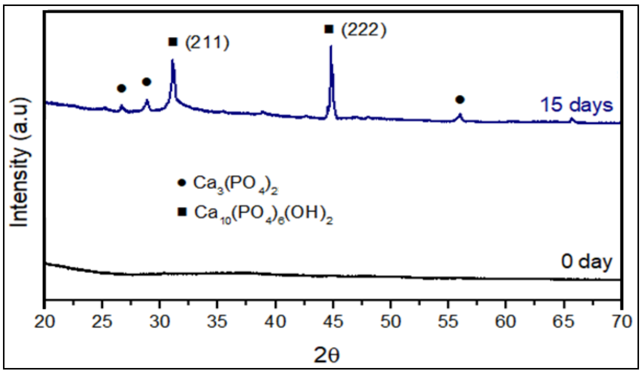

Fig 1: XRD diagram of bioactive glass $40SiO_2$-40CaO-$20P_2O_5$ before and after 15 days in SBF solution

Sensory analysis of toothpastes

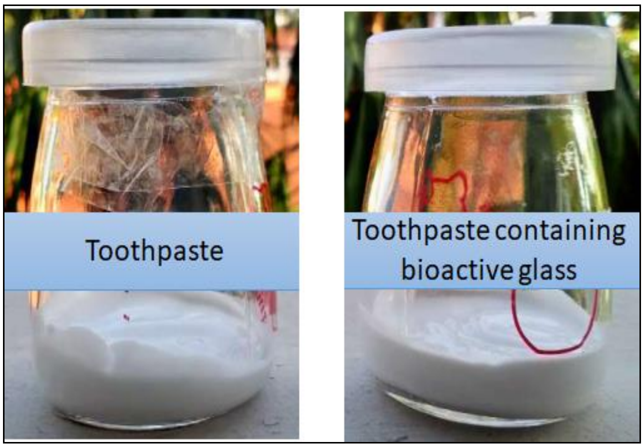

Fig 2: Images of toothpaste and toothpaste containing bioactive glass

Toothpaste and toothpaste products containing 5 wt.% by weight of bioactive glass were synthesized and stored in the glass jars as shown in Fig. 2. In terms of sensory food evaluation, both toothpaste and toothpaste containing bioactive glass are homogeneous pastes; pure white in color; with mild minty aroma, and slightly sweet taste of glycerin. There is no sensory difference when comparing the two types of synthetic toothpaste. The pH values of toothpaste and toothpaste containing bioactive glass are 7.86 and 7.92, respectively. When leaving the toothpaste samples at 45 °C for 2 days, the toothpaste samples were still quite homogeneous, with some air bubbles appearing, not drying,

Still retaining the characteristic aroma

Microbiological evaluation

Toothpaste and toothpaste containing bioactive glass products were also tested for common bacteria such as: Coliforms; Escherichia Coli; Staphylococcus Aureus. Obtained results showed that both toothpaste samples were immune to these bacteria (Tab. 1; Analyses effectuated at Center of analytical services and experimentation HCMC, dated 04/04/2019). Thus, synthetic toothpaste products are sensory-appropriate and safe from common harmful bacteria. These synthetic products can be used for everyday brushing.

Table 1: Microbiological test for toothpaste samples

Toothpaste			
No	**Parameters**	**Results**	**Method**
1	Coliforms	Not detected	Ref. ISO 4831:2006
2	Escherichia coli	Not detected	Ref. ISO 21150:2015
3	Staphylococcus	Not detected	Ref. ISO 22718:2015
Toothpaste containing bioactive glass			
No	**Parameters**	**Results**	**Method**
1	Coliforms	Not detected	Ref. ISO 4831:2006
2	Escherichia coli	Not detected	Ref. ISO 21150:2015
3	Staphylococcus	Not detected	Ref. ISO 22718:2015

Defect-recovery evaluation

Figure 3 shows the original tooth sample (left), and the same tooth sample that was brushed with the toothpaste containing bioactive glass for 1 month (right). Through visual observation, it can be noticed that the tooth sample brushed with the toothpaste containing bioactive glass is brighter when compared to the original tooth surface.

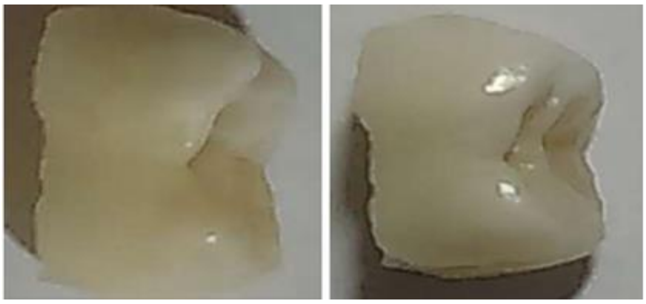

Fig 3: Photographs of tooth samples: (left) the original tooth sample and (right) the tooth sample brushed with the toothpaste containing bioactive glass after 1 month

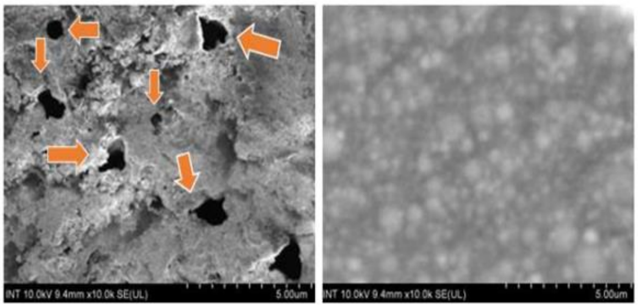

Fig 4: Surface SEM images of (left) primary teeth and (right) brushed teeth for 1 month using toothpaste containing bioactive glass

For more accurate assessment, primary tooth sample, and tooth sample brushed with the toothpaste containing bioactive glass were observed by SEM method as shown in Fig. 4. The initial tooth sample shows defects with different shapes on the tooth surface (left image) while the tooth sample brushed with toothpaste containing bioactive glass for 1 month has filled surface. The observation confirmed that the toothpaste containing bioactive glass is capable of restoring tooth defects by creating a new mineral apatite layer. The surface apatite formation can fill the defects, and make the tooth surface more even. The obtained result confirms the effectiveness of adding bioactive glass to toothpaste, and the bioactivity of added glass created a mineral layer that fills the tooth surface defects.

Conclusion

The bioactive glass material system $40SiO_2$-$40CaO$-$20P_2O_5$ was synthesized by sol-gel method. XRD analysis showed that the glass material has an intrinsically amorphous structure. *In vitro* experiment confirmed the bioactivity of synthetic glass through the formation of a new bone mineral layer hydroxyapatite (HA) after 15 days of soaking in SBF solution. The synthetic glass was added to the toothpaste to create a new product, with the function of restoring tooth defects thanks to its bioactive property. The obtained result showed a good recovery of tooth defects after tooth sample brushed with the toothpaste containing bioactive glass after a month. Thus, adding bioactive glass to toothpaste can create a new, quality product in tooth protection thanks to its bioactivity.

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