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RESEARCH ARTICLE

EFFECT OF Ag-DOPED ON PROPERTIES OF BIOACTIVE GLASS 45S.

Xuan Vuong Bui[1,2*]

1. Ton Duc Thang, University, Vietnam
2. HCM City Industry and Trade College

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Abstract

The objective of this paper is to synthesize the bioglass 45S doped with Ag (45S-Ag). The influence of doping Ag on the glass matrix was highlighted by DTA and XRD methods. The presence of Ag element was controled by EDX analysis. ‘‘In vitro’’ of synthesized glass was effectuated by soaking of glass powder in SBF solution. EDX result indicated that silver was released when immersing derivative bioglass in SBF solution and silver is an antibacterial agent. XRD and SEM confirmed the bioactivity of glass 45S-Ag by the apatite formation on its surface.

Introduction:-

The first bioglass was discovered by L. L. Hench. It named bioglass 45S with chemical composition $45SiO_2$-$24.5CaO$-$24.5Na_2O$-$6P_2O_5$ and synthesizedby melting method.It was used as animplant material in the human body to repair and replace diseased or damaged bone. Its bioactivity based on the ability to form a hydroxyapatite layer: $Ca_6(PO_4)_{10}(OH)_2$(HA) on the surface when immersing in a physiological solution or implanted in the human body. The formation of apatite layer promotes the adhesion of bone tissues and permits an intimate bone-bonding with the implants. Consequently, the bone architecture is repaired and reconstructed [1-2]. After the L. L. Hench’s discovery, many derivativebioglasses have been elaborated and estimated. That has opened up potential applications of bioglass material.

In recent years, the scientists are looking towards developing new bioactive materials doped with the silver element. In these biomaterials, the silver is considered as a bioactive agent. It plays an important role to limit the bacterial activity on biomaterials, resulting in the improvement of biological properties [3-4].

This work aim to synthesize the bioglass 45S doped with Ag by melting method. The percentage of Ag_2O (0,1 wt%) was incorporated into the glass (synthetic glass noted 45S-Ag). Some analysis techniques such as DTA, XRD, EDX, and SEM were used to investigate the synthesized biomaterial.

Experimental methods:-

Synthesis of bioglass 45S doped with Ag (45S-Ag):-

The original bioglass of L. L. Hench is 45S ($45SiO_2$ – $24.5CaO$ – $24.5Na_2O$ – $6P_2O_5$ wt%). In this study, bioglass 45S doped with 0.1 wt% of Ag_2O (45S-Ag) was synthesized by melting of a powder mixture of $CaSiO_3$, Na_2SiO_3, $Na_3P_3O_9$and Ag_2O at 1400 ^oC during 3 hours. At high temperature,Ag_2O was diffused and Ag^+replaced the positions

Corresponding Author:-Xuan Vuong Bui.
Address:-.Ton duc Thang, University, Vietnam.

of Na^+ and Ca^{2+} ions in the structure of bioglass.The obtained bulk glasses were ground into powder and sieved to achieve the bio-glass particles with size less than 100µm.
Chemical reactions at high temperature are below:

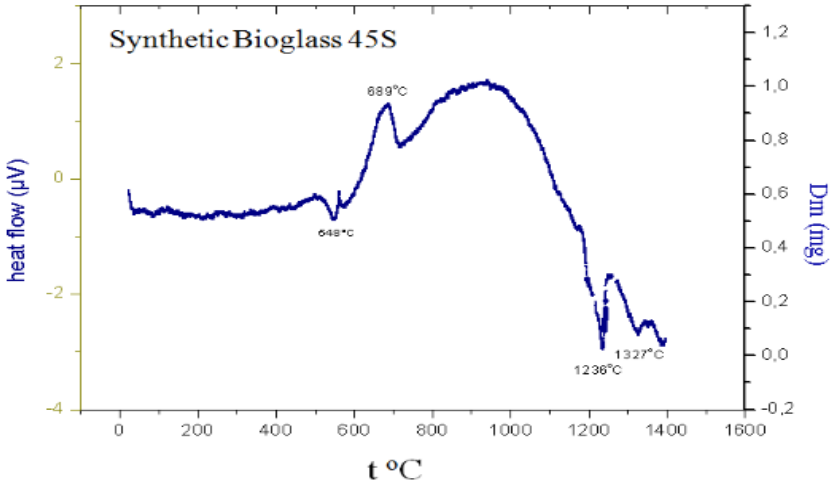
$$CaSiO_3 \rightarrow CaO + SiO_2$$
$$Na_2SiO_3 \rightarrow Na_2O + SiO_2$$
$$(NaPO_3)_3 \rightarrow 3/2\ Na_2O + 3/2\ P_2O_5$$

In vitro experiment:
''In vitro'' bioactivity of 45S-Ag wasinvestigated by soaking 100 mg of powdered samples with 200 ml of simulated body fluid (SBF). The SBF solutionwas prepared bythe method which is reported by Kokubo et al [5-6]. SBF solution has similar characteristics of pH, and chemical composition to human blood plasma. Immersion were maintained at body temperature (37°C), and agitation (50 tours/min) during 0, 3, 7 and 15 days. Then the glass powders were removed and rinsed with deionized water to stop the exchange reactions, and continuously rinsed absolute alcohol. After that the powder samples were dried and stored for further investigation of the formation of HA layer.

Physico-chemical characterizations:-
Differential thermal analysis (DTA)was used to provide date onthe transformations thathave occurred,such as glass transitions, crystallization and melting point of derivative glass.In order to characterize the amorphous character of synthetic bioglass and evaluate the formation of apatite layer after ''in vitro'' assays, X-ray diffraction (XRD) measurements were carriedon Bruker D8 Advancediffractometer. The XRD data were acquired in the range of 10-70° (2θ) with a scanning speed of 1°/min.Scanning Electron Microscopy (SEM) (Model JSM-6301, JEOL) was used to evaluate the morphological surface of synthetic bioglass before and after immersion in the SBF solution.Energy dispersive X-ray (EDX) was used to analyse the elemental presence in biomaterial.

Results and Discussion:-

DTA analysis:-



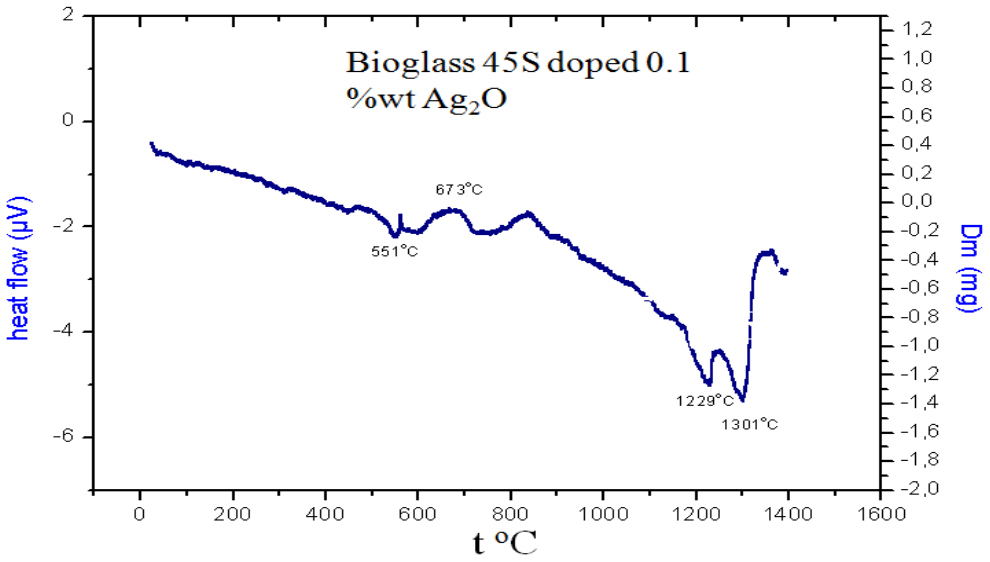


Figure 1:- DTA data of bioglass 45S and 45S-Ag

Figure 1 presents the DTA analyses of bioglass 45S and 45S-Ag. Obtained data showed an increase of glass transition temperature when glass doped with Ag_2O. While, crystallization, and fusion temperatures were decreased (Table 1). This result confirmed the effect of Ag from Ag_2O on the glassy matrix of bioglass 45S.

Table 1:- Temperature dataof 45S and 45S-Ag

Materials	**Temperature data**(^oC)			
	Glass transition temperature t_t	Crystallizationtemperature t_c	Fusion temperatures t_{f1} and t_{f2}	
45S	548	689	1236	1327
45S-Ag	551	673	1229	1301

XRD analysis:

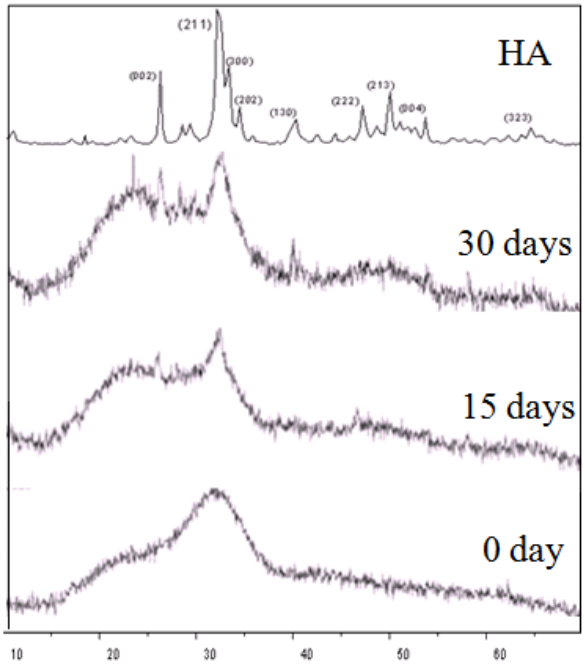


Figure 2:- Bioglass 45S-Ag before and after ‘‘In vitro’’ experiment

X-ray diffractogram of 45S-Ag showed a diffraction halo which is characteristic of the amorphous material. No peaks of Ag_2O or Ag could be observed in initial glass (Fig. 2). This result confirmed that the silver elements or Ag_2O compounds did not exist independently and they incorporated in the glassy matrix. It is considered that Ca^{2+}, Na^+ ions can be replaced by Ag^+ ions in the structure of bioglass 45S. In more detail, one Na^+ is equivalent to one Ag^+ and one Ca^{2+} correspond to two Ag^+ ions (Fig. 3).

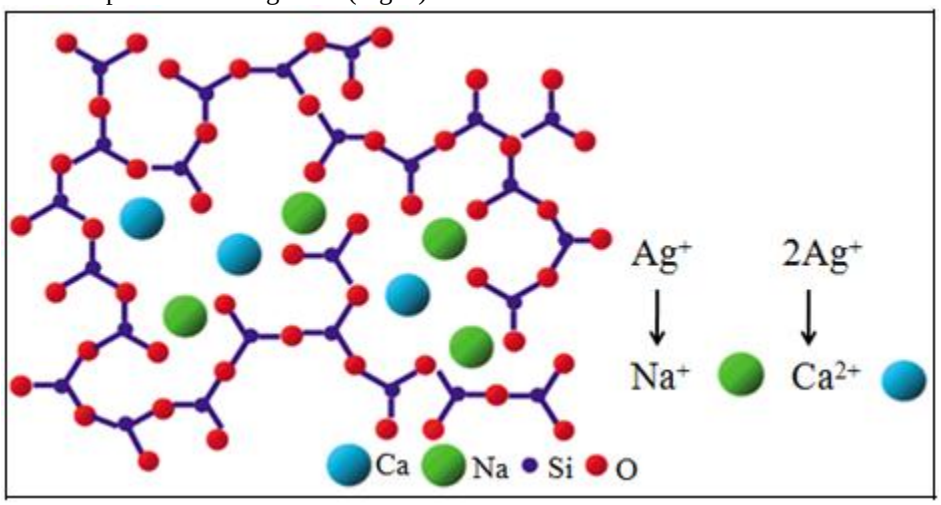


Figure 3:- Elemental structure of synthesized bioglass

After 15 and 30 days of immersion in SBF solution, XRD diagrams of glass 45S-Ag presented the sharp peaks corresponding to the hydroxyapatite (HA) phase[7-8] (Fig. 2). The formation of a new apatite layer on the glass surface illustrated the bioactivity of bioglass doped with 0.1 wt% of Ag_2O.

Energy Dispersive X-Ray Analysis (EDX):-

EDX result strongly confirmed the presence of silver in derivative bioglass (Table 2).After 30 days soaking in SBF fluid, the Ag concentration was zero (Table 3). This highlighted the release of Ag element from derivative glass to SBF solution during immersion times. The Ag^+ ions play an important role as an antibacterial agent. So, when this derivative bioglass is inserted into human body, it can damage to bacteria.

Table 2:- EDX analysis of bioglass 45S-Ag initial

Element	% mass	% atom
O	42.79	57.94
Na	14.81	13.95
Si	21.26	16.39
P	2.21	1.55
Ca	18.75	10.13
Ag	0.18	0.04
Total	100%	100%

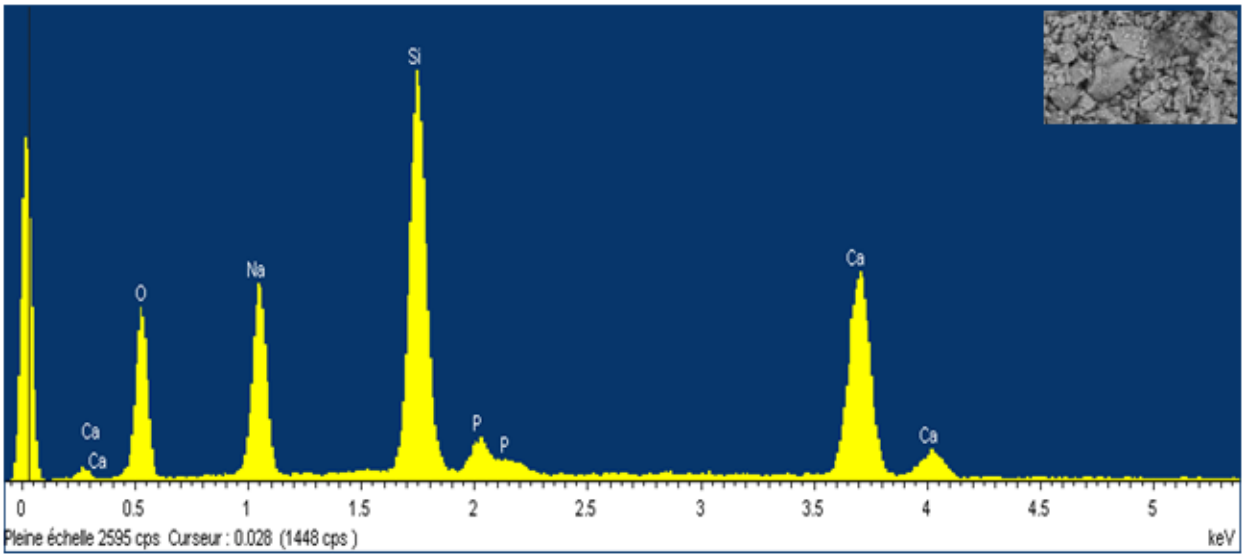
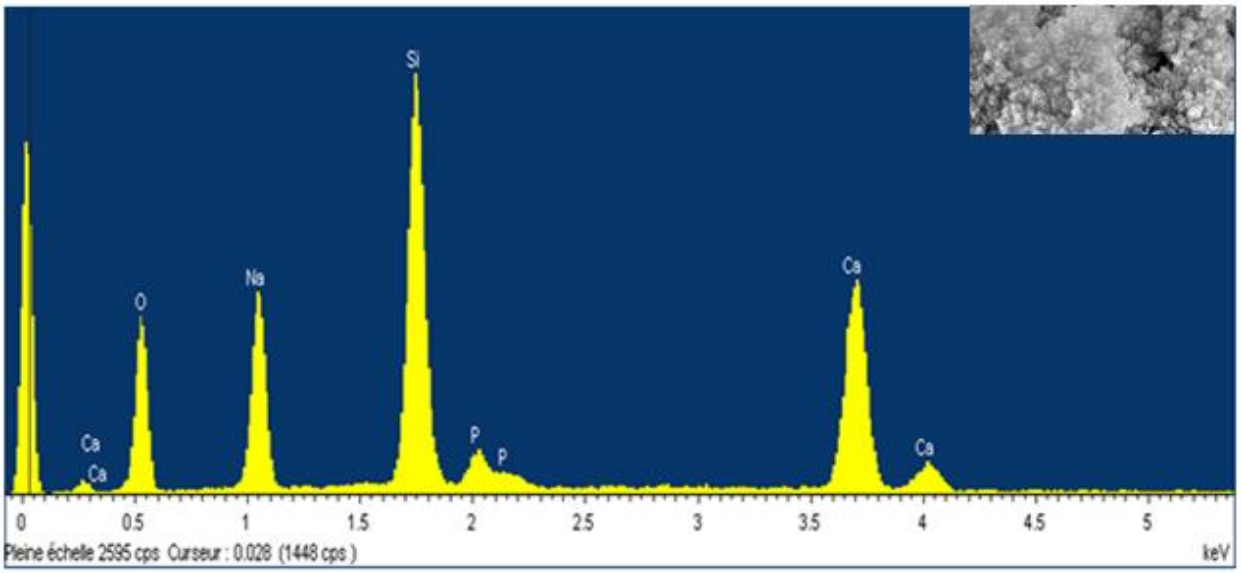


Table 3:- EDX analysis of bioglass 45S-Agafter 30 days of immersion.

Element	% mass	% atom
O	50.07	65.88
Na	3.60	3.30
Si	25.19	18.88
P	5.35	3.64
Ca	15.83	8.31
Ag	-0.03	-0.01
Total	100%	100%



SEM analysis:-

SEM images of bioglass 45S-Agwere presented in Fig. 3. They highlighted the change of surface morphologies when glass samples were dipped in SBF solution. According to the XRD analysis, this change attributed to the formation of a new apatite layer on the glassy surface (Fig. 4).

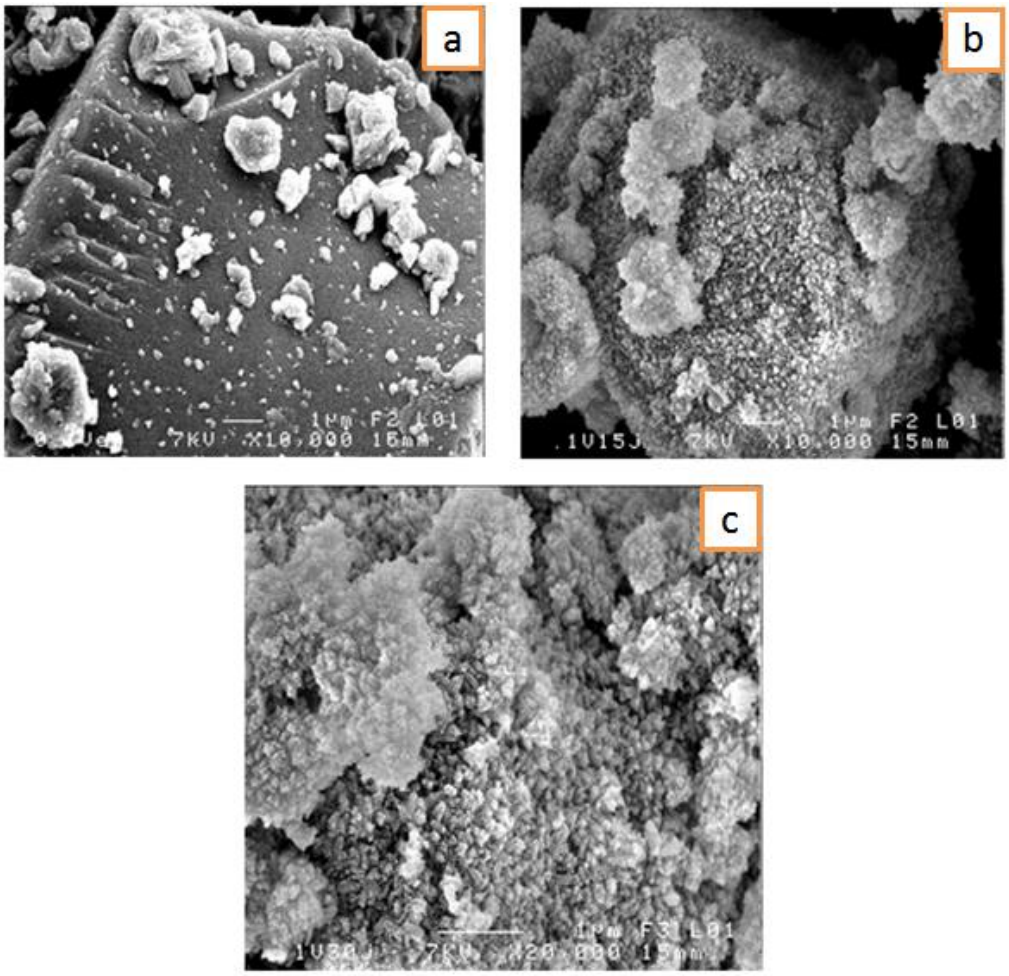


Figure 4:- SEM images of glass 45S-Ag before and after immersion: a) glass initial, b) glass after 15 days and c) glass after 30 days of immersion

Conclusions:-

We are synthesized successfully derivative bioglass, silver doped precursor bioglass 45S. DTA showed the effect of Ag on the character temperatures of bioglass.XRD and EDX results strongly confirmed the presence of silver in original bioglass 45S matrix. EDX results indicated that silver was released when immersing derivative bioglass in SBF solution and silver is an antibacterial agent. XRD and SEM confirmed the bioactivity of bioglass 45S-Ag. So, derivative bioglass still keeps its initial bioactivity characteristics. This demonstrated that silver doped bioglass 45S is a potential biomaterial.

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